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(71) Applicants (for all designated States except US):
RECKITT BENCKISER (UK) LIMITED [GB/GB]; 67 Alma Road, Windsor, Berkshire SL4 3HD (GB). **AQUASOL LIMITED** [GB/GB]; Writtle College Campus, Chelmsford, Essex CM1 3WN (GB).

(72) Inventors; and

(75) Inventors/Applicants (for US only): **DUFFIELD, Paul, John** [GB/GB]; 128 Canada Drive, Cherry Burton HU17

7RH (GB). **HAMMOND, Geoffrey, Robert** [GB/GB]; 574 James Reckitt Avenue, Hull, East Yorkshire HU8 0LG (GB). **EDWARDS, David, Brian** [GB/GB]; 88 Hayfield, Chells Manor, Stevenage, Hertfordshire SG2 7JR (GB). **MCCARTHY, William, John** [GB/GB]; 1 Sunnybank Cottages, Donhead St. Mary, Shaftesbury, Dorset SP7 9DL (GB). **BECKETT, Arnold, Heyworth** [GB/GB]; 20 Braybrooke Gardens, Fox Hill, Upper Norwood, London SE19 2UN (GB). **JACKMAN, Anthony, Douglas** [GB/GB]; 14 Thorley Gardens, Pyrford, Woking, Surrey GU22 8UL (GB).

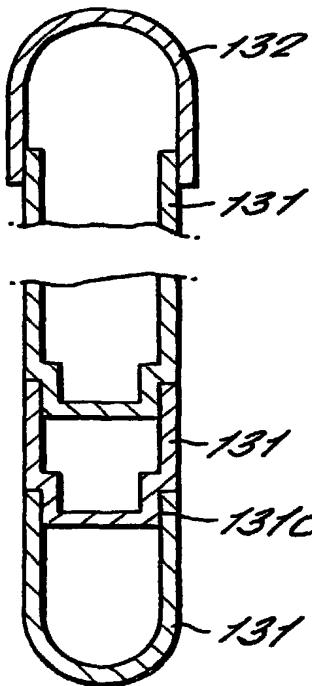
(74) Agent: **DICKSON, Elizabeth, Anne**; Reckitt Benckiser plc, Group Patents Dept., Dansom Lane, Hull HU8 7DS (GB).

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(54) Title: INJECTION-MOULDED WATER-SOLUBLE CONTAINER



(57) Abstract: The present invention provides a moulded rigid, water-soluble container (2, 111, 131, 141) with at least two components of which a substantial part of the surface of these components is water soluble or water dispersible so as to leave perforations throughout the wall when the capsular container (2, 111, 131, 141) is placed in contact with an aqueous environment, wherein the container (2, 111, 131, 141) has one to six compartments, preferably one or two or three, the accessibility time of the various compartments being the same or different from one compartment to another compartment.

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INJECTION-MOULDED WATER-SOLUBLE CONTAINER

The present invention relates to rigid, water-soluble containers. It also relates to capsules, in particular to capsules that may be utilised for the delivery into Man or other animals of substances such as ingestible ingredients like pharmaceutically- or nutritionally-active materials, that dissolve or disperse within the gastro-intestinal tract, and to capsule-like containers, in particular to such containers that may be utilised for the delivery into an aqueous environment of substances such as detergents, pesticides, biocides, deodorants, dyes and pigments, and water-treatment chemicals.

15

Clothes washing compositions may be delivered to a clothes washing machine by a delivery tray from which the composition is fed into the washing drum, or they may be placed directly into the washing drum. The washing compositions may be in powder, liquid or block form. Liquid compositions have the disadvantage that they may be spilt. The same applies to powder compositions. Powder compositions have the additional disadvantage that they may produce dust which can be inhaled. These problems are overcome or lessened when blocks of washing composition are used. These are normally individually wrapped. On unwrapping a block, for use, it is still possible that some dust may be produced. Additionally it is an inconvenience for the consumer to have to unwrap the block. Furthermore it is almost impossible for the user to avoid some contact between the block and his or her skin, so leading to a requirement for the user to

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wash their hands after starting the washing machine. In fact, all of the methods described involve a risk of contact between the composition and the skin, and it is desirable in all cases for the user to wash their hands 5 after starting the washing machine. In this context it should be borne in mind that many compositions contain enzymes to assist the cleaning action. Even though the user may tolerate enzyme residues which may be left in clothes after washing, they may still not tolerate 10 contact between the concentrated washing composition containing the enzymes, and the skin.

Similar considerations apply in relation to other areas including fabric care, surface care and 15 dishwashing. Thus, in relation in particular to dishwashing compositions, there are also problems of spillage, dust generation, skin contact and inconvenience.

20 It is known to package chemical compositions which may be of a hazardous or irritant nature in water-soluble or water-dispersible materials such as films. The package can simply be added to water in order to dissolve or disperse the contents of the package into the water.

25

For example, WO 89/12587 discloses a package which comprises an envelope of a water-soluble or water-dispersible material which comprises a flexible wall and a water-soluble or water-dispersible heat seal. The 30 package may contain an organic liquid comprising, for example, a pesticide, fungicide, insecticide or herbicide.

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CA-A-1,112,534 discloses a packet made of a water-soluble material in film form enclosing within it a paste-form, automatic dishwasher-compatible detergent 5 composition. The water-soluble material may be, for example, poly(vinyl alcohol), polyethylene oxide or methyl cellulose.

It is also known to form water-soluble containers by 10 thermoforming a water-soluble material. For example, WO 92/17382 discloses a package containing an agrochemical such as a pesticide comprising a first sheet of non-planar water-soluble or water-dispersible material and a second sheet of water-soluble or water-dispersible 15 material superposed on the first sheet and sealed to it by a continuous closed water-soluble or water-dispersible seal along a continuous region of the superposed sheets.

The above methods of packaging have, however, a 20 number of disadvantages.

The first disadvantage is that they do not have a particularly attractive appearance. In fields such as containers used in the domestic environment, an 25 attractive appearance for an article is extremely desirable. Liquids contained in envelopes of water-soluble film can have a limp, unattractive appearance.

The second disadvantage is that it is difficult to 30 form two or more separate compartments in the packaging so that two incompatible components are both enclosed but separated from each other. Although an arrangement has

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been described to separate incompatible materials in flexible pouches in WO 93/08095, the method proposed is complex and is not currently achievable in large-scale manufacturing. It cannot, therefore, be used for 5 producing large numbers of containers.

The third disadvantage is that there is only limited control of the release profile of the compositions held in the containers. For example, when a composition is 10 held between two planar water-soluble films or in a thermoformed package, the composition is simply released at the time when the films dissolve or disperse in water. While it may be possible to control to a certain extent the timing of the start of release of the contents, there 15 can be no control over the rate of release of the contents since the entire film dissolves or disperses at about the same time. Furthermore it can be difficult to provide an extended time before the contents of the package are released. An additional problem also arises 20 with thermoformed packages. If the thermoforming is not carefully controlled there may be inadvertent thinning of the film material at the points where the material is drawn down into the mould when it is thermoformed. This could release the contents of the package early. 25 Additionally, in all of the above packages, it is not possible to release different compositions at different times or at different rates since, as discussed above, it is not possible to incorporate more than one composition in each water-soluble container.

30

The fourth disadvantage is that the containers cannot be produced at a particularly fast rate. When the

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containers are produced by heat-sealing planar films or by thermoforming, the containers have to be immediately filled and sealed. All of these procedures have to be carried out in succession. This means that it is not 5 possible to obtain a quick throughput for mass-market goods such as household products. For example, standard thermoforming machines can only produce around 400 to 800 containers per minute.

10 There are numerous forms of systems used in the delivery of medical preparations in the market place today. The two most dominant in relation to oral routes are capsules made from hard gelatine, and tablets - the so-called solid dose formulations. Both of these 15 presentations have remained virtually unchanged for decades. Gelatine capsules are made by a dipping process, building up successive layers, while tablets are formed by compressing a powder or fine granules.

20 The gelatine capsules currently employed are used extensively throughout the world to deliver thousands of prescribed and over-the-counter medications and nutritional formulations. Unfortunately, they have a number of highly significant limitations, these 25 including: their inability easily to be formed into a shape that facilitates the optimum delivery of their ingredients into the Patient; the fact that gelatine is animal-based; and the substantial likelihood of them sticking in the Patient's oesophagus when they are 30 swallowed. In recent years these and other limitations - see below - have been acknowledged, and efforts have been made to overcome them by finding and using a number of

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materials as alternatives to gelatine, but in most cases the materials are even more brittle, more difficult to shape, and significantly more expensive than gelatine and other conventional solid dose delivery systems, and 5 therefore they have not thus far been used successfully for this purpose - which leaves the problem of the hard gelatine capsule, and its disadvantages, still to be solved. Some of these disadvantages are as follows:

10 As noted above, gelatine is animal-based, being extracted from bones and hides, and as such it carries the risk - or, at least, the perceived risk - of being linked with Creutzfeldt-Jakob disease. The manufacturing process used to make hard gelatine capsules involves a 15 so-called dipping process, which makes thickness parameters difficult to control. More significantly, the process does not lend itself to the more complex shapes, sizes and chemical characteristics now required within the pharmaceutical and nutraceutical industries, more 20 specifically when controlled release is desirable. Hard gelatine capsules also have an inherent problem of attracting a static charge, which makes their handling during manufacture an additional problem, while the gelatine itself has a tendency to undergo detrimental 25 physical and chemical changes during long-term storage.

As also pointed out above, gelatine capsules may be rather hard to swallow properly, for they can all too easily stick in the oesophagus. Now, this may seem 30 trivial, but in fact whilst the most frequent cause of accidents to Patients in hospitals is falling out of bed, the second most frequent cause is capsules or tablets

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sticking in the Patient's oesophagus! Very few Patients are able to swallow a capsule when lying down, and when a gelatine capsule sticks in the oesophagus it can be extraordinarily difficult to dislodge. Indeed, it has 5 been shown that drinking liquids such as water fails to move such a stuck capsule even when taking large amounts, and on occasion even eating food fails to overcome the adhesion. Part of the problem may be that a filled gelatine capsule will float if its contents are not dense 10 (as is often the case), and will have a tendency to remain in the mouth, after the initial mouthful of water has been swallowed. This allows stickiness rapidly to develop on the surface of the capsule, which in turn increases the probability that the capsule will stick in 15 the oesophagus when finally swallowed.

It has now been appreciated that the above type of capsule has utilisations other than in medicine and the human or animal body. In particular, it has been 20 realised that many substances that must be packaged for delivery to their use site could, where that site is an aqueous environment, be contained in similar, though somewhat larger, capsules. Thus, a capsule-like container - a "capsular" container - could be employed to 25 deliver, for example, detergents to a washing machine, pesticides to a paddy field, or water-treatment chemicals to a reservoir. Moreover, by appropriately dimensioning the various parts of the container, or by suitably selecting the materials from which they are made, 30 different parts of the container will in use dissolve at different times.

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The present invention seeks to provide water-soluble containers which overcome some or all of the above disadvantages.

5 The present invention has a number of different aspects and embodiments as follows:

10 The present invention provides a rigid, water-soluble container made of an injection moulded polymer, for example, a poly(vinyl alcohol) (PVOH) and/or a cellulose ether such as hydroxypropylmethylcellulose (HPMC), which container encases a composition, for example, a fabric care, surface care or dishwashing composition.

15 The present invention also provides a capsule, i.e. a container, comprising a self-supporting receptacle part and a closure part, the receptacle part and the closure part together enclosing a composition, for example a fabric care, surface care or dishwashing composition, the receptacle part being formed of a water-soluble polymer, and the closure part being formed of a water-soluble polymer, wherein, in use, the closure part dissolves before the receptacle part.

25 The present invention additionally provides an injection-moulded capsule container of any size or shape for the delivery of a water-destined ingredient, preferably selected from a fabric care, surface care or dishwashing composition, which container is made of a material that will dissolve in the intended aqueous destination site.

The present invention further provides a method of ware washing, comprising use of a container, receptacle or washing capsule as defined above, the method entailing 5 introducing the container, receptacle or washing capsule into a ware washing machine prior to commencement of the washing process, the container, receptacle or washing capsule being entirely consumed during the washing process. The ware washing machine may, for example, be a 10 dishwashing or laundry washing machine.

The present invention also provides a capsule container comprising at least two components made of one or more material(s) that can be moulded and which are 15 water soluble or water dispersible or in which a substantial part of the surface of these components is water soluble or water dispersible so as to leave perforations throughout the wall when the capsular container is placed in contact with an aqueous environment, wherein the container has one to six 20 compartments, preferably one or two or three, the content of the various compartments being accessible to the aqueous environment when the capsular container is exposed to such an aqueous environment, the accessibility 25 time of the various compartments being the same or different from one compartment to another compartment. The content of the container may, for example, not be a fabric care, surface care or dishwashing composition.

30 The following description and drawings all relate to each and every aspect and embodiment as discussed above, either singly or in any combination thereof.

The containers of the present invention overcome some or all of the above disadvantages.

5 Firstly, because the containers are rigid and self-supporting, they have an attractive, uniform appearance which does not vary between different containers. Furthermore, the rigid containers can easily have various elements incorporated which are considered to be pleasing
10 to the eye but which are impossible to incorporate in the flexible containers discussed above.

15 Secondly, because the containers are rigid, it is easily possible to introduce two or more compartments, or have larger compartments separated by walls, to separate mutually incompatible ingredients. The containers can also hold part of the composition on an external surface, for example in an indentation. Furthermore, the container can be moulded in almost any shape that might
20 be useful. In particular it can be given raised or lowered areas.

25 Thirdly, it is possible to control the release profile of the contents of the container. Since the container is rigid, it is possible to adapt the width of all of the walls of the container to control both the start of release of the composition as well as the rate of release. For example, one or more walls may be made thin in order to have an early release of the
30 composition. Alternatively all the walls may be thick in order to ensure that there is a delayed release of the composition. The rate of release of the composition may

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also be controlled by ensuring that only part of the container has thin walls which are dissolved or dispersed before the remainder of the container. Different walls or parts of walls of the container may be prepared from 5 different water-soluble polymers which have different dissolution characteristics. For example, a first compartment may be fully enclosed by a polymer such as PVOH which dissolves at a higher or lower temperature than the polymer enclosing a second compartment. Thus 10 different components can be released at different times. If the container holds a solid or gelled composition, it is not even necessary for the container to fully enclose the composition. A part may be left exposed, so that it immediately begins to dissolve when added to water.

15

Fourthly, since the containers are rigid and self-supporting, they can easily be filled on a production line using normal filling equipment. Such filling equipment is quite capable of filling at least 1500 20 containers per minute.

Desirably the container, apart from its contents, consists essentially of the injection-moulded polymer. It is possible for suitable additives such as 25 plasticizers and lubricants to be included. Plasticizers are generally used in an amount of up to 20 wt%, for example from 15 to 20 wt%, lubricants are generally used in an amount of 0.5 to 5% wt% and the polymer is generally therefore used in an amount of 75 to 84.5 wt%, 30 based on the total amount of the moulding composition. Examples of suitable polymers are PVOH and cellulose ethers such as HPMC.

5 PVOH is a known water-soluble material which is used to prepare water-soluble films for encasing compositions as discussed above. Cellulose ethers have not in general been used to prepare water-soluble films because they have poor mechanical strength.

10 PVOH materials, unlike gelatin, can be modified to dissolve at different rates under various conditions (including the pH of the aqueous medium into which they are introduced).

15 The PVOH preferably used to form the container of the present invention may be partially or fully alcoholised or hydrolysed. For example it may be from 40-100%, preferably 70-92 %, more preferably about 88%, alcoholised or hydrolysed polyvinylacetate. The polymer such as PVOH or cellulose ether is generally cold water (20°C) soluble, but may be insoluble in cold water at 20°C and only become soluble in warm water or hot water having a temperature of, for example, 30°C, 40°C, 50°C or even 60°C. This parameter is determined in the case of PVOH by its degree of hydrolysis.

25 For certain applications or uses, polymers soluble in aqueous environments at temperatures as low as 5°C are also desirable.

30 In order to ensure that the polymer such as PVOH or cellulose ether is capable of being injection moulded, it is usual to incorporate components such as plasticizers and mould release agents in an amount of up to, for

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example, 15 wt% of the composition. Suitable plasticizers are, for example, pentaerthritol such as depentaerythritol, sorbitol, mannitol, glycerine and glycols such as glycerol, ethylene glycol and 5 polyethylene glycol.

Solids such as talc, stearic acid, magnesium stearate, silicon dioxide, zinc stearate, and colloidal silica may also be used. A preferred PVOH which is 10 already in a form suitable for injection moulding is sold in the form of granules under the name CP1210T05 by Soltec Developpement SA of Paris, France.

The PVOH may be moulded at temperatures of, for 15 example, from 180-220°C, depending upon the formulation selected and the melt flow index required. It can be moulded into containers, capsule bodies, caps, receptacles and closures of the appropriate hardness, texture and solubility characteristics.

20

One of the great practical problems of current hard gelatine capsules is their ability to hold a static electrical charge. Such capsules in production rapidly pick up a high static charge which has the effect of 25 making them not only stick to each other and to all other non-polar surfaces but also making them attract particles of foreign material from their surroundings. It also means that that the capsules are hard to fill, and that their surfaces must be treated immediately prior to 30 printing. This phenomenon is common to some mouldable polymers, but not to PVOH, which is not only soluble, ingestible, mouldable and weldable, but in addition will

not support a static charge capable of causing the problems described above. So, yet another consequence of using an injection-moulding method is that the mouldable material may be chosen having regard to its ability to 5 pick up and retain a static charge - or may include one or more additional substances that has some effect on the way the capsule behaves in this respect.

Thus, in a still further aspect this invention 10 provides an injection-moulded container such as a receptacle or capsular container made from materials that will not hold a static charge, such as PVOH or a cellulose ether.

15 One aspect of the present invention is, as indicated above, a capsule, i.e. a container, comprising a self-supporting receptacle part and a closure part, the receptacle part and the closure part together enclosing a composition such as a fabric care, surface care or 20 dishwashing composition, the receptacle part being formed of a water-soluble polymer, and the closure part being formed of a water-soluble polymer, wherein in use, the closure part dissolves before the receptacle part.

25 Preferably the capsule is a washing capsule enclosing a washing composition.

Another aspect of the present invention is, as indicated above, an injection-moulded capsule container 30 of any size or shape for delivery of a water-destined ingredient, in particular selected from a fabric care, surface care or dishwashing composition, a detergent,

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pesticide, biocide, deodorant, dye, pigment or water-treatment chemical, which container is made of a material that will dissolve in the intended aqueous destination site.

5

In many aspects of the present invention, including these aspects, the water-soluble polymer is not limited to PVOH or a cellulose ether. Other water-soluble compounds may be used, such as polyglycolides, gelatine, 10 polylactides and polylactide-polyglycolide copolymers. These components may also, if necessary, contain components such as plasticizers and mould release agents, such as those described above. All of the polymer compositions, including the PVOH and cellulose ether, may 15 also include other components such as colouring agents and components which modify their properties.

In all aspects and embodiments of the present invention, the container or capsule generally comprises a 20 receptacle part which holds the composition and a closure part, which may simply close the receptacle part or may itself have at least some receptacle function. The receptacle part preferably has side walls which terminate at their upper end in an outward flange in which the 25 closure part is sealingly secured, especially if the closure part is in the form of a film. The securement may be by means of an adhesive but is preferably achieved by means of a seal, between the flange and the closure part. Heat sealing may be used or other methods such as 30 infra-red, radio frequency, ultrasonic, laser, solvent, vibration or spin welding. An adhesive such as an

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aqueous solution of PVOH or a cellulose ether may also be used. The seal is desirably also water-soluble.

5 The closure part may itself be injection moulded or blow moulded. Preferably, however, it is a plastics film secured over the receptacle part. The film may, for example, comprise PVOH or a cellulose ether such as HPMC or another water-soluble polymer.

10 The container walls have thicknesses such that the containers are rigid. For example, the outside walls and any inside walls which have been injection moulded independently have a thickness of greater than 100 μm , for example greater than 150 μm or greater than 200 μm , 300 μm , 15 or 500 μm , 750 μm or 1mm. Preferably, the closure part is of a thinner material than the receptacle part. Thus, typically, the closure part is of thickness in the range 10 to 200 μm , preferably 50 to 100 μm , and the wall thickness of the receptacle part is in the range 300 to 20 1500 μm , preferably 500 to 1000 μm . The closure part may, however, also have a wall thickness of 300 to 1500 μm , such as 500 to 1000 μm .

25 Preferably, the closure part dissolves in water (at least to the extent of allowing the washing composition in the receptacle part to be dissolved by the water; and preferably completely) at 40°C in less than 5 minutes, preferably in less than 2 minutes.

30 The receptacle part and the closure part could be of the same thickness or different thicknesses. The closure

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part may, for example, be of higher solubility than the receptacle part, in order to dissolve more quickly.

5 Preferably, the washing capsule is generally cuboid in its external shape, with the top wall being formed by the closure part, and with the side walls and base wall being formed by the receptacle part.

10 Preferably, a washing capsule of the invention is manufactured by forming an array of receptacle parts, each receptacle part being joined to adjacent receptacle parts, and being separable from them by a snap or tear action. The array is preferably one which has columns and rows of the receptacle parts. The receptacle parts 15 may be separated by frangible webs of the water-soluble polymer such as PVOH or a cellulose ether.

20 Alternatively, the receptacle parts may be manufactured with the aforementioned flanges, such that they are separated from each other by a line of weakness. For example the material may be thinner, and so able to be broken or torn readily. The thinness may be a result of the moulding process or, preferably, of a later scoring step.

25

30 In the manufacturing method, the array, formed by injection moulding, is fed to a filling zone, and all the receptacle parts are charged with the washing composition. A sheet of a water-soluble polymer such as PVOH or a cellulose ether may then be secured over the top of the array, to form the closure parts for all the receptacle parts of the array. The array may then be

split up into the individual washing capsules, prior to packaging, or it may be left as an array, for packaging, to be split by the user. Preferably, it is left as an array, for the user to break or tear off the individual 5 washing capsules. Preferably, the array has a line of symmetry extending between capsules, and the two halves of the array are folded together, about that line of symmetry, so that closure parts are in face-to-face contact. This helps to protect the closure parts from 10 any damage, between factory and user. It will be appreciated that the closure parts are more prone to damage than the receptacle parts. Alternatively two identical arrays of washing capsules may be placed together with their closure parts in face-to-face 15 contact, for packaging.

In some embodiments of the invention the container, capsule or receptacle part may define a single compartment. In other embodiments of the invention the 20 container, capsule or receptacle part may define two or more compartments, which contain different products useful in a washing process. In such a situation a dividing wall or walls of the compartments preferably terminate at the top of the container, capsule or 25 receptacle part i.e. in the same plane as the top edges of the side walls, so that when the receptacle part is closed by the closure part the contents of the compartments cannot mix. The container, capsule or receptacle part may be provided with an upstand, 30 preferably spaced from the side walls thereof, and preferably of generally cylindrical shape. If wished, the remaining volume of the container, capsule or

receptacle part can be divided into two or more parts by means of walls extending between the upstand and the side walls.

5 The container, capsule, receptacle part or closure may be formed with an opening, for example a depression, formed in the side wall or the base wall, and preferably being open in the outward direction. That is to say, it preferably does not form part of the main volume defined
10 by the container, capsule, receptacle part or closure. Preferably the opening is adapted to receive, in a press-fit manner, a solid block (for example a tablet) of a composition, for example a material useful in a washing process.

15 Preferably, the closure part is of a transparent or translucent material, so that the contents of the washing capsule can be seen.

20 Preferably, the container, capsule or receptacle part is of a transparent or translucent material, so that the contents of the washing capsule can be seen.

25 The washing composition within the container, capsule or receptacle part, or within a compartment thereof, need not be uniform. For example during manufacture it could be fed first with a settable agent, for example a gel, useful in a washing process, and then with a different material. The first material could
30 dissolve slowly in the washing process so as to deliver its charge over a long period within the washing process. This might be useful, for example, to provide immediate,

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delayed or sustained delivery of a softening agent in a clothes washing container, capsule or a receptacle part.

The container, or capsule may, for example, be in at 5 least two parts (a body part and a cap part) which fit tightly, and preferably sealingly and inseparably, together to form a compartment in which is stored the ingredient to be achieved. In one example, the container or capsule may have three parts - a body such as a 10 receptacle, a first cap, and then a second cap to fit over the closed end of either the body or the first cap, so as to result in a capsule with two separate compartments. Where there are three such parts (or more; four parts - a body and three caps - make three 15 compartments, and so on), then naturally the ingredients in each compartment may be the same or they may be different.

In all embodiments of the present invention one 20 compartment may contain, for example, a liquid or solid component (such as a powder, granules or a compressed or gelled tablet) and another may contain a different liquid or solid component (such as a powder, granules or a compressed or gelled tablet). Alternatively, more than 25 one component may be present in one or more compartments. For example a compartment may contain a solid component, for example in the form of a ball or pill (such as a powder, granules or a compressed or gelled tablet), and a liquid component.

30

By using container, receptacle or capsule cap/body parts of different thicknesses, or of different polymers,

or both, such as PVOH polymers with different degrees of hydrolysis, this invention enables enhanced control over the release of different ingredients at different times or in different positions within broad scope of the 5 aqueous destination.

The capsular container can be of any size or shape. It is, for example, conveniently of the standard capsule shape - an elongate tubular package with closed, rounded 10 ends. Moreover, although it is possible to have the several parts of much the same sizes, it is usual that there will be a long body with a shorter cap (the cap may be half or a quarter the length of the body). Typically, a capsular container has an overall closed length of 4 to 15 10 cm, such as 4 to 6 cm, and an external diameter of 2 to 4 cm. However, it should be understood that there is no theoretical limitation, in either size or shape, and what is suitable will normally be decided upon the basis of the "dose" of the container's contents, the size of 20 any aperture the container may have to pass through, and the available means of delivery.

The capsular container may be in at least two parts (a body or receptacle part and a cap part) which fit 25 tightly, and preferably sealingly and inseparably, together. The actual joining of the parts can be carried out in any convenient way, but advantage can be taken of the very nature of the capsule material - that fact that it is one that can be injection-moulded (it is a 30 thermoplastic). Thus, the preferred joining method is welding, for example either heat welding, by melting the parts when they are in contact, and allowing them to

"run" into each other and then cool and solidify to become an integral device, or solvent welding, where much the same effect is achieved by partially dissolving the adjacent portions of the capsule and letting them again 5 run into each other and then solidify to form a whole. Heat welding is much the preferred way, although any of the sealing techniques described herein may be used.

Indeed, in one of its several aspects the invention 10 specifically provides an injection-moulded capsular contained having a cap portion and a body portion which, after filling, are welded together into a single indivisible unit (so sealing in and preventing subsequent access to the contents, and thus ensuring containment of 15 the contents, whether solid, powder, granular, liquid, gel or suspension presentations).

In another aspect, this invention provides a capsule 20 that may be utilised for the delivery of some active ingredient or device into the human or animal body, which capsule is made of a material that can be injection-moulded and will at least in part dissolve in the body.

The invention provides a capsule - that is to say, a 25 small container for the relevant ingredients, which container is in at least two parts (a body part and a cap part) which fit tightly, and preferably sealingly and inseparably, together to form a compartment in which is stored the ingredient to be delivered. As an 30 alternative, the capsule may have three parts - a body, a first cap, and then a second cap to fit over the closed end of either the body or the first cap, so as to result

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in a capsule with two separate compartments. And where there are three such parts (or more; four parts - a body and three caps - make three compartments, and so on), then naturally the ingredients in each compartment may be
5 the same or they may be different.

In one example - see Figure 11A in the accompanying Drawings - the capsule may have a body and cap each provided with a central axially-parallel partition, so
10 that the capsule as a whole has two separate compartments.

By using capsule cap/body parts of different thicknesses, or of different polymers, or both, this
15 invention enables enhanced control over the release of different active ingredients at different times or in different positions. This difference in release time is useful in many applications or uses including within the gastro-intestinal tract, in which the ability to control
20 release time is of utility in the developing science of chrono-biology.

The capsule is of any shape, preferably an elongate tubular package. The ends are advantageously closed,
25 whether rounded or conical. Moreover, although it is possible to have the several parts of much the same sizes, it is usual that there will be a long body with a shorter cap (the cap may be half or a quarter the length of the body). Typically, a capsule has an overall closed
30 length of 10-25mm and an external diameter of 5-10mm for pharmaceutical or nutraceutical use.

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Although it is possible to have the several parts of much the same sizes, it is usual that there will be a long body with a shorter cap (the cap may be half or a quarter the length of the body). Typically, a capsular 5 container for applications or uses other than pharmaceutical or nutraceuticals has an overall closed length of 3 to 12 cm, for example 4 to 10 cm and an external diameter of 1 to 5 cm, for example 2 to 4 cm. However, it should be understood that there is no 10 theoretical limitation, in either size or shape, and what is suitable will normally be decided upon the basis of the "dose" of the container's contents, the size of any aperture the container may have to pass through, and the available means of delivery.

15

The invention's capsule is intended to be utilised for the delivery of some active ingredient or device into the human or animal body. The delivery may be by any appropriate route; for most active ingredients the oral 20 route is preferred - and it is when the capsule is administered orally that its advantages are most apparent - but rectal or vaginal routes may of course be employed if appropriate. Regardless of the nature of the route, however, it is clearly necessary that the material from 25 which the capsule is made - the material that can be injection-moulded - should of course be safe for delivery into the target organism (which may be a Human or some other animal). PVOH - polyvinylalcohol - is such a material; not only is it non-toxic but it is available in 30 food-quality grades, and it is very much preferred.

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PVOH - or more specifically PVOH-based formulations - is presently the most convenient injection-mouldable, water-soluble or water-dispersible, material, and of the various commercially-available PVOH formulations one 5 particularly-preferred variety is that range of materials sold (in the form of granules) under the name CP1210T05 by Soltec Developpement SA of Paris, France

In general, PVOH polymers are synthetic materials 10 capable, when appropriately formulated with other adjuvants - such as plasticisers, particularly glycerine (but other glycols and polyglycols may be used depending upon their acceptability for ingestion), and solids such as talc, stearic acid, magnesium stearate, silicon 15 dioxide, zinc stearate, and colloidal silica - of being moulded at temperatures between 180-220°C, depending upon the formulation selected and the melt flow index required, into capsule bodies and caps of the appropriate hardness, texture and solubility characteristics required 20 of a pharmaceutical or like capsule.

PVOH materials, unlike gelatine, can be modified to dissolve at different rates under varying conditions (including the pH of the aqueous medium - such as the 25 interior parts of the target organism's body - into which they are introduced). Capsules made from PVOH materials can therefore be formulated to release their contents in any desirable location, for example, as far as pharmaceutical use is concerned, in the stomach, the 30 upper or lower small intestine, or the colon, as considered desirable.

Furthermore, PVOH formulations generally do not interact with many organic solvents or oils of the type used in pharmaceutical or nutraceutical compositions, while the aqueous gels often utilised in such 5 compositions can be formulated to resist interaction with PVOH, so that capsules made from PVOH can be used to contain such materials.

The invention provides a capsule which is in at 10 least two parts (a body part and a cap part) which fit tightly, and preferably sealingly and inseparably, together. The actual joining of the parts can be carried out in any convenient way, but advantage can be taken of the very nature of the capsule material - the fact that 15 it is one that can be injection-moulded (it is a thermoplastic). Thus, the preferred joining method is welding - either heat welding, by melting the parts when they are in contact, and allowing them to "run" into each other and then cool and solidify to become an integral 20 device, or solvent welding, where much the same effect is achieved by partially dissolving the adjacent portions of the capsule and letting them again run into each other and then solidify to form a whole. Heat welding is much the preferred way.

25

Indeed, in one of its several aspects the invention specifically provides an injection-moulded capsule (suitable for use in the delivery of some active ingredient or device) having a cap portion and a body 30 portion which, after filling, are welded together into a single indivisible unit (so sealing in and preventing subsequent access to the contents, and thus ensuring

containment of the contents, whether granular, liquid, gel or suspension presentations).

PVOH materials are particularly suited to thermal 5 welding, a convenient variety of this technique being laser welding, though any suitable method can be used providing it does indeed make a permanent weld with the polymer used to form the capsule. Some other common methods are infra-red (IR), radio frequency (RF), and 10 ultrasonic welding.

Some of these methods may require the addition of other items or processes to ensure their correct 15 operation. For example, RF welding may require the use of a metal (normally aluminium) conductor in contact with the capsule surface. Laser welding will normally require the top surface to be transparent to the laser used, and the lower surface to be opaque to it. This can be achieved by avoiding opaque coatings and fillers on the 20 outer surface of the capsule cap and by their application to the outer surface of the capsule body. For example, a circumferential line of a suitable material can be printed around the body at the required joining point to facilitate the weld at that point. As a result of the 25 welding, a circumferential weld situation on a planar cross-section of the capsular container is advantageously obtained.

Of the various methods, the laser weld is preferred 30 as there is no direct contact required, and it can achieve the very high production speeds required.

After placing the intended contents in the capsule body, and putting the cap on the body, the two portions of the capsule can be welded - by means of a laser beam, say - into a single unit which cannot thereafter readily 5 and without leaving visible traces be separated into body and cap in order to gain access to the contents. Accordingly, any attempt to tamper with the contents would be clearly obvious.

10 The two parts of the capsule that are to be welded together are, for example, made so that the open end of one will pass into the open end of the other with the smallest gap that can be practically achieved to allow easy assembly. Normally, but not necessarily, the 15 capsule is designed with a stop on one or other component so that the entry of one into the other cannot overrun and stops at the same fixed position in every case.

20 The two halves or shells are in the closed position when the entire periphery of the open end of one is overlapped by the periphery of the open end of the other. The closed capsule is then ready for welding, and this is done by bringing the capsule into close proximity to the welding head. This distance will vary with the method of 25 welding chosen. The welding equipment is operated, and forms a weld between the two layers in contact in the form of a line of weld in a closed loop around the periphery of the capsule. This can be achieved either by having the welding heads in the form of a ring (which may 30 be continuous or made up of a number of discrete heads), or by rotating one or other of the capsule and the head around the other - say, by rolling the capsule past the

head. The exact method will depend on the welding technology chosen.

It is also possible to use solvent welding - that is, using a solvent for the chosen injection-mouldable material so as to soften and render flowable the surface layers of the material where the two parts are in contact. In the PVOH case the solvent is conveniently water or an aqueous electrolyte solution (typically containing an alkali metal halide such as lithium chloride as the electrolyte). This technique, however, requires another stage to the welding process, in which the solvent is applied to one of the surfaces to be in contact before the two shells are closed. This method is not preferred, however, as it is likely to be comparatively slow, and the addition of water and solute may well be detrimental to the ingredient(s) or other preparations contained within the capsule.

The weldability of the two parts (body and cap) of the injection-moulded capsule of the invention into a single unit which cannot subsequently be separated into its two parts without visibly destroying the capsule is in contrast to the nature of the known hard gelatine capsule parts, which cannot be so welded. Thus, the integrity of the contents can be protected by the invention's capsule in a way which cannot take place using capsule parts made of gelatine.

Due to the integrity of the welded seal, in all aspects and embodiments the container, receptacle or

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capsule can be filled with any appropriate powder, liquid, gel, or oil.

The invention provides a capsule, container or receptacle made of a material that can be injection-moulded. The injection-moulding process allows controlled variations in the thickness of the walls and domed ends of either or both halves of the capsule, thereby allowing the release characteristics to be infinitely varied. The use of such moulded capsule shells permits the development of capsule formulations containing controlled-release beads or granules which can be determined where the contents are released so that the system as a whole can be made to deliver its contents at the desired position, rate and period of release irrespective of differing physioco-chemical properties of the contents. This also enables the delivery system to be used to protect the drug against adverse conditions in other parts of the organism - the gastro-intestinal tract, for example - before absorption occurs if the capsule or container is intended for administration to the human or animal body.

There are many advantages in the production of capsules using injection-moulding as compared with the traditional dip-coating methods, and it is worth setting out a few here.

Dip-coating of gelatine is the traditional method for the production of capsule shells. One of the principal properties of a capsule is the rate at which the shell material dissolves or disperses to release the

contained ingredients. Using the dipping process there is only a limited control over the final thickness of the capsule shell. The principal advantage of using the injection-moulding process is that there is much greater

5 versatility over the final component form, for example:-

a) The thickness of the wall sections can be more closely controlled, and hence may be varied inter alia to obtain the appropriate dissolution rate of the capsule.

10 b) Reduced wall thickness possible with injection-moulded capsule shells will result in increased production rates.

c) The surface form (smoothness) of both inner and outer capsule surfaces can be more closely controlled for moulded as compared with dipping, which latter only allows control of the inner

15 surface form.

d) The degree (tightness) of fit between the two capsule halves can be more closely controlled with moulding.

20 e) Injection-moulding permits the addition of sectional variation around the rim of either or both of the capsule halves, so that features for final capsule assembly, such as ultrasonic or laser welding, can be included in the basic component design.

25 f) If both capsule halves are moulded simultaneously, in the same injection-mould tool, the capsule halves can be assembled automatically as a post-moulding operation carried out immediately the tool halves

30 open (with benefits for cleanliness and quality assurance).

g) There are no requirements for further trimming or sizing operations.

The invention provides a capsule for the delivery
5 into the human or animal body of an active ingredient or device. For the most part the ingredient will, as suggested hereinbefore, be a drug - a pharmaceutically-active substance - or perhaps some sort of nutritionally-active material - a "nutraceutically-active" material -
10 such as vitamins or oligo-elements or food supplements. However, it is not impossible for this capsule to be used for the delivery of quite a different sort of "ingredient" - for example, a measuring or sampling device, or machine, as might be required in some forms of
15 medicine or surgery.

In its broadest aspect this invention provides a capsule made of a material that can be injection-moulded. This injection-moulding concept has several unexpected
20 consequences, as does the choice of a polymer of the PVOH type for this purpose. Specifically, an injection-moulded capsule can be moulded in almost any shape that might be useful (as might have been inferred from what has been said above). In particular, it can be given
25 external raised (or lowered) areas - this has the advantage that, for the preferred orally delivery route, it significantly reduces the surface area of the capsule that is able to come into contact with the walls of the oesophagus as the capsule is being swallowed, and thereby
30 reduces the risk of the capsule sticking in the oesophagus, and thus facilitates the passage of the capsule down into the stomach.

In another aspect, therefore, the invention provides an injection-moulded capsule (suitable for use in the delivery of some active ingredient or device) having 5 raised portions moulded into its external surface.

Thus the container, capsule, capsular container, receptacle or closure may, for example, have raised portions moulded into its external surface.

10

The raised portions - for the most part they are referred to hereinafter as "raised", though obviously the effect of a raised part can be achieved by lowering the other parts - can be in the form of short, small pimple-like projections, or they can be ribs that extend wholly or partially either around or along the capsule. The portions may be designed to include or act as markings allowing identification of the capsule and its contents - either visually, by the sighted, or tactilely, by the 15 visually-impaired, or even by a machine or reader. Thus a code can be moulded into the surface so that a filled capsule can be identified at all stages of its life - by the manufacturer for quality assurance and quality 20 control, by a wholesaler or retailer as part of a stock-control system, and by the user before utilisation, 25 particularly those with vision impairment.

The surface of the capsule, container, receptacle or closure needs no pre-treatment prior to printing.

30

By suitable cutting of the moulds used, any required pattern can be moulded into the surface, either raised or

incuse. Both raised and incuse variants bring different properties to the capsule, and the benefits of each are described hereinafter. The complexity of the pattern is limited only by the practical limitations on mould
5 making.

Thinner areas of the walls of different compartments of the capsular container are preferably disposed longitudinally according to the general elongated shape
10 of the capsular container.

The use of an incuse pattern has a number of interesting possibilities. For example, for sparingly-soluble drugs delivered orally, the gastro-intestinal
15 transit from mouth to rectum is often too short to allow the active ingredient of some orally-delivered medicament to be absorbed, with the consequence that most of the drug is excreted, and so wasted. However, incuse moulding in a suitable pattern provides a way of
20 converting the capsule - in, say, the acidic conditions prevailing in the stomach - from an integral, sealed, container to a perforate container from which the contents of the capsule can readily escape as a solution or suspension (rather like a tea bag, or a metal tea
25 infuser).

Such an incuse pattern design may include a capsule of standard form but with relatively thick walls. Around a suitable section of the capsule is moulded an array of
30 thin-walled incuse panels. Once the capsule has reached the stomach, the thin-walled panels in the capsule body quickly dissolve, leaving the capsule with a grid

structure of holes. These holes can be small enough to prevent the internal contents leaving the capsule, but large enough to allow the dissolving medium to enter and make contact with the contents of the capsule. As has 5 been described earlier, PVOH materials can, due to variations in molecular weight and extent of hydrolysis, be selected to dissolve at different speeds and at different temperatures in aqueous conditions. Hence, by varying the thickness and the dissolution characteristics 10 of the injection-moulded capsule materials, the body of the capsule may be designed to dissolve or break up at a chosen rate especially in the stomach. Once the capsule has dissolved or broken up, the beads or granules are released but only after being retained in the stomach for 15 an extended period of up to 12 hours. As long as such capsules with holes remain intact, they do not pass through the pyloric sphincter into the duodenum until the housekeeper wave is in operation.

20 More generally for applications or uses outside of washing, the difference of accessibility time to an aqueous environment from one compartment to another is in the range of 1 minute to 12 hours at the same temperature in the range of 5°C to 95°C.

25 Another possibility is to mould a capsule in a relatively sparingly-soluble polymer material - such as a high molecular weight PVOH having a high degree of hydrolysis - with a similar array of holes (rather than 30 thin-walled soluble panels), and then in a separate process, after filling and capping, to cover the area containing the holes with a relatively soluble polymer

either by spraying or by shrinking or gluing a soluble sleeve thereover. It should be noted that in use such a "covered" perforate capsule may either break up in the gastro-intestinal tract after being swept from the 5 stomach, thereby releasing its ingredients, or it may carry on to leave the body in the faeces while still containing the active-ingredient-carrying beads or granules (though these have by then been relieved of most of the active-ingredient content). The relatively- 10 sparingly soluble polymer used in this case could even be an insoluble polymer - provided, of course, that it is both injection-mouldable and tolerated by the body.

By this means, such a capsule of outer diameter 15 between 3 and 6 mm may contain, for example, a plurality of beads slightly larger than the holes which will be formed in the capsule and on which the finely-divided sparingly-soluble drug is layered. The drug dissolves only slowly in the acid conditions prevailing in the 20 stomach. The capsule, because of its size, can be retained in the stomach and thus allow the release in solution form of the drug for absorption in the stomach and gastro-intestinal tract. In this way, the absorption of the sparingly soluble drug in the gastro-intestinal 25 tract will be increased as the beads are held for a longer time in the stomach than they would be if released from a gelatine capsule that rapidly dissolves with the result that the beads pass quickly from the stomach into the small intestine. In the "fed state", units of 30 dimensions greater than 3mm do not pass through the pyloric sphincter into the duodenum as long as there are contents in the stomach. Thus, if such a PVOH capsule is

taken with the breakfast meal, it will be retained in the stomach until after the evening meal if a normal midday meal was taken. If the capsule has not dissolved or broken up in the stomach, it will be swept from the 5 stomach into the large intestine where it may either dissolve or break up or be eliminated from the body in the faeces. The overall result is an increased transit time of the drug delivery system from mouth to faeces, and thus increased bio-availability for sparingly-soluble 10 drugs.

The capsule which either contains or develops holes while keeping its integrity can also be used advantageously to retain in the stomach beads containing 15 soluble drugs and possessing controlled-release membranes programmed to take advantage of the better absorption of such drugs in the small intestine rather than the large intestine, and thereby to give a constant rate of systemic drug input.

20

These hole-containing or hole-developing capsules can be used to release two or more drugs at designated regions each at a controlled relative rate even if the drugs in conventional form have different rates of drug 25 absorption or metabolism in different regions of the gastro-intestinal tract.

Whilst the oral route is preferred for many of the drug applications envisaged using the capsules of the 30 invention, the rectal and vaginal routes, particularly those utilising perforate capsules which produce holes *in vivo*, are also important.

The oral route is suitable generally for sparingly-soluble drugs, and for good control of drug input and activation location.

5

The rectal route is particularly appropriate for use with perforate capsules that produce holes *in vivo*, together with controlled-release drug-carrying beads or granules. This allows the avoidance of "first pass metabolism" - some drugs are especially sensitive to this when administered orally. The perforate capsule can deliver the drug at a controlled rate *via* its location in the rectum so that the drug, unlike the delivery from a suppository, is released locally from the beads or granules in the capsule to give a steady, localised, input into the lower haemorrhoidal vein (unlike the higher medium and upper haemorrhoidal veins, which deliver blood to the liver, this allows systemic delivery without "first pass metabolism" by the liver). If they were not contained in the capsule, the beads would move upwards into the descending colon, and so would supply their drug content mainly to the medial and upper haemorrhoidal veins. Thus, using the rectal route with a perforate capsule, a drug can be delivered to a Patient in a similar but more acceptable manner to that achieved by intravenous infusion.

The vaginal route with a perforate capsule facilitates drug delivery at a constant rate followed by cessation when the system is withdrawn from the body aperture at the designated time.

From the above examples, other more selective approaches can be developed to maximise and control the rate of drug input by the chosen route of product use, thereby offering solutions to many current problems of 5 drug delivery in man and other animals.

Another consequence of using an injection-moulding method is that the mouldable material may easily include one or more additional substance that has some effect on 10 the way the capsule behaves in use - for instance, on its surface properties (and specifically on its tackiness, or stickiness), or on its rate of dissolution.

Thus, in yet another aspect the invention provides 15 an injection-moulded capsule (suitable for use in the oral delivery of some active ingredient or device) that is made from an injection-mouldable material that contains one or more particulate hydrophobic solid in order to both reduce the surface tackiness and also 20 increase the density of the capsule, which effects will reduce the risk of the capsule sticking in the oesophagus.

This meets one of the problems of current hard 25 gelatine capsules - and of those made of any other water soluble polymer - namely that upon insertion in the mouth the capsule comes in contact with water, which will begin the softening process prior to dissolving and lead to a stickiness of the surface which can cause problems and 30 interruptions (sometimes leading to release of its contents in the oesophagus) on the capsule's path through the oesophagus to the stomach. As noted, reduction of

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this stickiness can be achieved by modifying the mouldable polymer formulation by the addition of inert solids in powder form - though naturally the added solids have to be approved for ingestion, and must be compatible with 5 the medical preparation contained within the capsule.

This use of added solids provides a more rigid capsule shell with a surface less immediately affected by the aqueous content of the mouth or oesophagus, thereby 10 reducing surface tackiness during the initial swallowing.

In this aspect - the incorporation of a particulate solid to influence tackiness - the solid is very preferably extremely finely divided, typical particle 15 sizes being in the range 1-50 micron, and preferably 5-10 micron. The upper limit is generally a practical one for the moulding process, but with increasing solid particle size the capsule surface will be to a greater extent made up of the insoluble solid ingredient and to a 20 lesser extent the polymer (which will be partially concealed below the contact surface with the oesophagus).

Materials that can be utilised to reduce the capsule's surface tackiness are most preferably insoluble 25 and preferably hydrophobic. Substances suitable for this purpose are talc, stearic acid, magnesium stearate, zinc stearate, sodium stearate, colloidal silica and magnesium trisilicate, with talc and magnesium stearate being especially preferred.

30

And in still another aspect the invention provides an injection-moulded capsule (suitable for use in the

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oral delivery of some active ingredient or device) that is made from an injection-mouldable material that contains one or more particulate solid in order to accelerate the rate of dissolution of the capsule, for 5 example in the different conditions of pH which exist in the gastro-intestinal tract.

Unlike gelatine capsules, which sometimes release their contents prematurely (especially in elderly 10 patients) when they stick in the oesophagus and open, capsules of PVOH (in particular) can be formulated so that they do not open in the oesophagus but release their contents only where necessary - ie, when they reach the relevant target area.

15 The particulate solid incorporated into the injection mix may be a material that is barely affected in a non-acidic medium but dissolves relatively rapidly in an acidic environment, so as to allow the capsule to 20 release its contents, for example in the stomach. Alternatively, the solid material may be one that is relatively insoluble in an acidic medium but relatively soluble in a neutral environment, so as to allow release of the capsule's contents, for example in the lower small 25 intestine and in the colon.

The simple dissolution of the solid in the chosen medium is sufficient to cause a significant acceleration in the capsule break-up, particularly so when a gas is 30 also generated, when the physical agitation caused will result in the virtually immediate release of the contents from the capsule.

Such solids are of course subject to the same limitations of approval and compatibility as before. The solids which can be used for accelerating the rate of 5 dissolution of the capsular container are preferably the bicarbonate and carbonate salts of the alkali and alkaline-earth metals, typically sodium, potassium, magnesium and calcium, all of which salts may liberate carbon dioxide gas for the purpose of generating 10 effervescence.

The solid is very preferably extremely finely divided, typical particle sizes being in the range 1-25 micron, and preferably 5-10 micron.

15 Materials that can be utilised to affect the capsule's dissolution rate in a non-acid medium (for example, the lower intestine or the colon) but without being affected by an acid medium (for example, the stomach) are most preferably solid acidic substances with carboxylic or sulphonic acid groups or salts thereof. Substances suitable for this purpose are cinnamic acid, tartaric acid, mandelic acid, fumaric acid, maleic acid, malic acid, pamoic acid, citric acid, and naphthalene 20 disulphonic acid, as free acids or as their alkali or alkaline-earth metal salts, with tartaric acid, citric acid, and cinnamic acid in the form of acids or their 25 alkali metal salts being especially preferred.

30 One of the great practical problems of current hard gelatine capsules is their ability to hold a static electrical charge. Such capsules in production rapidly

pick up a high static charge which has the effect of making them not only stick to each other and to all other non-polar surfaces but also making them attract particles of foreign material from their surroundings. It also 5 means that the capsules are hard to fill, and that their surfaces must be treated immediately prior to printing.

This phenomenon is common to some mouldable polymers, but not to PVOH, which is not only soluble, 10 ingestible, mouldable and weldable, but in addition will not support a static charge capable of causing the problems described above. So, yet another consequence of using an injection-moulding method is that the mouldable material may be chosen having regard to its ability to 15 pick up and retain a static charge - or may include one or more additional substance that has some effect on the way the capsule behaves in this respect.

Thus, in a still further aspect this invention 20 provides an injection-moulded capsule (suitable for use in the delivery of an active ingredient or device into the human or animal body) being made from materials that will not hold a static charge.

25 The capsule of the invention is one that, utilised for the delivery of some active ingredient or device into the human or animal body, dissolves in the body to release its contents therein. The term "dissolve" is used herein in a fairly general sense, to indicate that 30 the capsule crumbles, decomposes, disintegrates or disperses; it need not actually dissolve, although most often it will.

Another possibility is to mould a capsule, container or receptacle in a relatively sparingly-soluble polymer material - such as a high molecular weight PVOH having a 5 high degree of hydrolysis - with a similar array of holes (rather than thin-walled soluble panels), and then in a separate process, after filling and capping, to cover the area containing the holes with a relatively soluble polymer either by spraying or by shrinking or gluing a 10 soluble sleeve thereover. The relatively-sparingly soluble polymer used in this case could even be an insoluble polymer - provided, of course, that it is injection-mouldable.

15 Another consequence of using an injection-moulding method is that the mouldable material may easily include one or more additional substance that has some effect on the way the capsule behaves in use - for instance, on its rate of dissolution.

20

Thus, in still another aspect the invention provide a container, for example, relatively-large injection-moulded capsular container, receptacle, capsule or closure that is made from an injection-mouldable material 25 that contains one or more particulate solid in order to accelerate the rate of dissolution of the container. This solid may also be present in the contents of the container, receptacle or capsule.

30 The simple dissolution of the solid in the chosen medium is sufficient to cause a significant acceleration in the container break-up, particularly so if a gas is .

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also generated, when the physical agitation caused will result in the virtually immediate release of the contents from the container.

5 The most obvious solids for this purpose are the bicarbonate and carbonate salts of the alkali and alkaline-earth metals, typically sodium, potassium, magnesium and calcium.

10 The solid is very preferably extremely finely divided, typical particle sizes being the range 1 to 25 μm , and preferably 5 to 10 μm .

15 Other materials that can be utilised to affect the capsule's dissolution rate are most preferably solid acidic substances with carboxylic or sulphonic acid groups or salts thereof. Substances suitable for this purpose are cinnamic acid, tartaric acid, mandelic acid, fumaric acid, maleic acid, malic acid, pamoic acid, 20 citric acid and naphthalene disulphonic acid, as free acids or as their alkali or alkaline-earth metal salts, with tartaric acid, citric acid, and cinnamic acid in the form of acids or their alkali metal salts being especially preferred.

25

 The container or capsule of the present invention may contain any composition which is intended to be released when the container is placed in an aqueous environment.

30

 Thus it may, for example, contain a fabric care, surface care or dishwashing composition. A fabric care

composition is any composition which is used in the field of fabric care, such as in a fabric washing, fabric treating or dyeing process. A surface care composition is any composition which is used in the field of surface care, for example to clean, treat or polish a surface. Suitable surfaces are, for example, household surfaces such as worktops, as well as surfaces of sanitary ware, such as sinks, basins and lavatories. A dishwashing composition is any composition which is used in the field of dishwashing, such as a dishwashing, water-softening or rinse aid composition.

Examples of such compositions are a dishwashing, water-softening, laundry, detergent and rinse-aid compositions. In this case the composition is especially suitable for use in a domestic washing machine such as a clothes washing machine or dishwashing machine. Other examples are disinfectant, antibacterial and antiseptic composition, for example those intended to be diluted with water before use, or a concentrated refill composition, for example for a trigger-type spray used in domestic situations. Such a composition can simply be added to water already held in the spray container.

The container may be used to contain any composition. Desirably the composition has a mass of at least 10 g or 15 g, for example, from 10 g or 15 g to 100 g, especially from 10 g to 15 g to 40 g. For example, a dishwashing composition may weigh from 10 g or 15 g to 20 g, a water-softening composition may weigh from 25 g to 35 g, and a laundry composition may weigh from 10 g to 40 g, 20 g to 40 g or 30 g to 40 g.

The container may also contain, for example, a detergent, pesticide, biocide, deodorant, dye, pigment or water-treatment chemical. It may, for example, deliver 5 detergents or water-treatment chemicals to a washing machine.

For pharmaceutical or nutraceutical applications or uses, the typical mass of the contents of the capsular 10 container is in the range of 10 mg to 15 g, preferably 50 mg to 1 g.

For uses other than pharmaceutical, nutraceutical or washing, the typical mass of the contents of the capsular 15 container is in the range of 1 g to 100 g, preferably 2 g to 50 g.

In general, particularly when used in a domestic environment, the maximum dimension of the container is 5 20 cm. For example, a cuboid container may have a length of 1 to 5 cm, especially 3.5 to 4.5 cm, a width of 1.5 to 3.5 cm, especially 2 to 3 cm, and a height of 1 to 2 cm, especially 1.25 to 1.75 cm.

25 The composition contained by the capsule may be, for example, any which is suitable for the designated application, for example a clothes washing or dishwashing application. It may be a powder or a liquid but if a liquid, may be a low water formulation, preferably having 30 a maximum water content of 5 wt%, in order to maintain the integrity of the walls of the capsule or a higher water formulation containing, for example, at least 8 wt%

water. The composition may be formulated having regard to the fact that the user will not come into contact with the composition, whether by inhalation or by skin contact. For example, the composition may include an 5 enzyme, without concern about physical contact between the composition containing the enzyme, and the user.

If the container contains an aqueous liquid having a relatively high water content, it may be necessary to 10 take steps to ensure the liquid does not attack the water-soluble polymer if it is soluble in cold water (20°C), or water at a temperature of up to, say, 35°C. Steps may be taken to treat the inside surfaces of the container, for example by coating it with agents such as 15 PVdC (poly(vinylidene chloride)) or PTFE (polytetrafluoroethylene), or to adapt the composition to ensure that it does not dissolve the polymer. For example, it has been found that ensuring the composition has a high ionic strength or contains an agent which 20 minimises water loss through the walls of the container will prevent the composition from dissolving the polymer from the inside. This is described in more detail in EP-A-518,689 and WO 97/27743.

25 The composition held within the container depends, of course, on the intended use of the composition. It may, for example, contain surface active agents such as an anionic, non-ionic, cationic, amphoteric or zwitterionic surface active agent or mixture thereof.

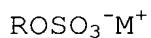
30

Examples of anionic surfactants are straight-chained or branched alkyl sulfates and alkyl polyalkoxylated

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sulfates, also known as alkyl ether sulfates. Such surfactants may be produced by the sulfation of higher C₈-C₂₀ fatty alcohols.

5 Examples of primary alkyl sulfate surfactants are those of formula:



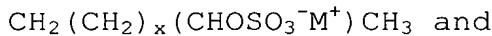
wherein R is a linear C₈-C₂₀ hydrocarbyl group and M is a water-solubilising cation. Preferably R is C₁₀-C₁₆ alkyl, 10 for example C₁₂-C₁₄, and M is alkali metal such as lithium, sodium or potassium.

15 Examples of secondary alkyl sulfate surfactants are those which have the sulfate moiety on a "backbone" of the molecule, for example those of formula:



wherein m and n are independently 2 or more, the sum of m+n typically being 6 to 20, for example 9 to 15, and M is a water-solubilising cation such as lithium, sodium or 20 potassium.

Especially preferred secondary alkyl sulfates are the (2,3) alkyl sulfate surfactants of formulae:



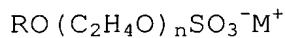
25



for the 2-sulfate and 3-sulfate, respectively. In these formulae x is at least 4, for example 6 to 20, preferably 30 10 to 16. M is cation, such as an alkali metal, for example lithium, sodium or potassium.

- 50 -

Examples of alkoxylated alkyl sulfates are ethoxylated alkyl sulfates of the formula:



5

wherein R is a C₈-C₂₀ alkyl group, preferably C₁₀-C₁₈ such as a C₁₂-C₁₆, n is at least 1, for example from 1 to 20, preferably 1 to 15, especially 1 to 6, and M is a salt-forming cation such as lithium, sodium, potassium, 10 ammonium, alkylammonium or alkanolammonium. These compounds can provide especially desirable fabric cleaning performance benefits when used in combination with alkyl sulfates.

15

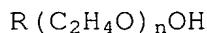
The alkyl sulfates and alkyl ether sulfates will generally be used in the form of mixtures comprising varying alkyl chain lengths and, if present, varying degrees of alkoxylation.

20

Other anionic surfactants which may be employed are salts of fatty acids, for example C₈-C₁₈ fatty acids, especially the sodium potassium or alkanolammonium salts, and alkyl, for example C₈-C₁₈, benzene sulfonates.

25

Examples of nonionic surfactants are fatty acid alkoxylates, such as fatty acid ethoxylates, especially those of formula:



30

wherein R is a straight or branched C₈-C₁₆ alkyl group, preferably a C₉-C₁₅, for example C₁₀-C₁₄, or C₁₂-C₁₄ alkyl

group and n is at least 1, for example from 1 to 16, preferably 2 to 12, more preferably 3 to 10.

5 The alkoxylated fatty alcohol nonionic surfactant will frequently have a hydrophilic-lipophilic balance (HLB) which ranges from 3 to 17, more preferably from 6 to 15, most preferably from 10 to 15.

10 Examples of fatty alcohol ethoxylates are those made from alcohols of 12 to 15 carbon atoms and which contain about 7 moles of ethylene oxide. Such materials are commercially marketed under the trademarks Neodol 25-7 and Neodol 23-6.5 by Shell Chemical Company. Other 15 useful Neodols include Neodol 1-5, an ethoxylated fatty alcohol averaging 11 carbon atoms in its alkyl chain with about 5 moles of ethylene oxide; Neodol 23-9, an ethoxylated primary C₁₂-C₁₃ alcohol having about 9 moles of ethylene oxide; and Neodol 91-10, an ethoxylated C₉-C₁₁ primary alcohol having about 10 moles of ethylene oxide.

20

Alcohol ethoxylates of this type have also been marketed by Shell Chemical Company under the Dobanol trademark. Dobanol 91-5 is an ethoxylated C₉-C₁₁ fatty alcohol with an average of 5 moles ethylene oxide and 25 Dobanol 25-7 is an ethoxylated C₁₂-C₁₅ fatty alcohol with an average of 7 moles of ethylene oxide per mole of fatty alcohol.

30 Other examples of suitable ethoxylated alcohol nonionic surfactants include Tergitol 15-S-7 and Tergitol 15-S-9, both of which are linear secondary alcohol ethoxylates available from Union Carbide Corporation.

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Tergitol 15-S-7 is a mixed ethoxylated product of a C₁₁-C₁₅ linear secondary alkanol with 7 moles of ethylene oxide and Tergitol 15-S-9 is the same but with 9 moles of ethylene oxide.

5

Other suitable alcohol ethoxylated nonionic surfactants are Neodol 45-11, which is a similar ethylene oxide condensation products of a fatty alcohol having 14-15 carbon atoms and the number of ethylene oxide groups per mole being about 11. Such products are also available from Shell Chemical Company.

10 Further nonionic surfactants are, for example, C₁₀-C₁₈ alkyl polyglycosides, such as C₁₂-C₁₆ alkyl polyglycosides, especially the polyglucosides. These are especially useful when high foaming compositions are desired. Further surfactants are polyhydroxy fatty acid amides, such as C₁₀-C₁₈ N-(3-methoxypropyl) glyciamides and ethylene oxide-propylene oxide block polymers of the 20 Pluronic type.

Examples of cationic surfactants are those of the quaternary ammonium type.

25 Examples of amphoteric surfactants are C₁₀-C₁₈ amine oxides and the C₁₂-C₁₈ betaines and sulfobetaines.

30 The total content of surfactants in the laundry or detergent composition is desirably 60 to 95 wt%, especially 75 to 90 wt%. Desirably an anionic surfactant is present in an amount of 50 to 75 wt%, the nonionic surfactant is present in an amount of 5 to 20 wt%, the

cationic surfactant is present in an amount of from 0 to 10 wt% and/or the amphoteric surfactant is present in the amount of from 0 to 10 wt%. These amounts are based on the total solids content of the composition, i.e.

5 excluding the water when present.

Dishwasher compositions usually comprise a detergency builder. Suitable builders are alkali metal or ammonium phosphates, polyphosphates, phosphonates, 10 polyphosphonates, carbonates, bicarbonates, borates, polyhydroxysulfonates, polyacetates, carboxylates such as citrates and other polycarboxylates. The builder is desirably present in an amount of up to 90 wt%, preferably 15 to 90 wt%, more preferably 15 to 75 wt%, 15 relative to the total weight of the composition. Further details of suitable components are given in, for example, EP-A-694,059, EP-A-518,720 and WO 99/06522.

The compositions, particularly when used as laundry 20 washing or dishwashing compositions, may also comprise enzymes, such as protease, lipase, amylase and cellulase enzymes. Such enzymes are commercially available and sold, for example, under the registered trade marks Esperase, Alcalase, Savinase, Termamyl, Lipolase and 25 Celluzyme by Nova Nordisk A/S. Desirably the enzymes are present in the composition in an amount of from 0.5 to 3 wt%, especially 1 to 2 wt%.

The compositions may, if desired, comprise a 30 thickening agent or gelling agent. Suitable thickeners are polyacrylate polymers such as those sold under the trade mark CARBOPOL, or the trade mark ACUSOL by Rohm and

Hass Company. Other suitable thickeners are xanthan gums. The thickener, if present, is generally present in an amount of from 0.2 to 4 wt%, especially 0.2 to 2 wt%.

5 The compositions can also optionally comprise one or more additional ingredients. These include conventional detergent composition components such as further surfactants, bleaches, bleach enhancing agents, builders, suds boosters or suds suppressors, anti-tarnish and anti-
10 corrosion agents, organic solvents, co-solvents, phase stabilisers, emulsifying agents, preservatives, soil suspending agents, soil release agents, germicides, phosphates such as sodium tripolyphosphate or potassium tripolyphosphate, pH adjusting agents or buffers, non-
15 builder alkalinity sources, chelating agents, clays such as smectite clays, enzyme stabilizers, anti-limescale agents, colourants, dyes, hydrotropes, dye transfer inhibiting agents, brighteners, and perfumes. If used, such optional ingredients will generally constitute no
20 more than 10 wt%, for example from 1 to 6 wt%, the total weight of the compositions.

25 The builders counteract the effects of calcium, or other ion, water hardness encountered during laundering or bleaching use of the compositions herein. Examples of such materials are citrate, succinate, malonate, carboxymethyl succinate, carboxylate, polycarboxylate and polyacetyl carboxylate salts, for example with alkali metal or alkaline earth metal cations, or the
30 corresponding free acids. Specific examples are sodium, potassium and lithium salts of oxydisuccinic acid, mellitic acid, benzene polycarboxylic acids, C₁₀-C₂₂ fatty

acids and citric acid. Other examples are organic phosphonate type sequestering agents such as those sold by Monsanto under the trade mark Dequest and alkylhydroxy phosphonates. Citrate salts and C₁₂-C₁₈ fatty acid soaps
5 are preferred.

Other suitable builders are polymers and copolymers known to have builder properties. For example, such materials include appropriate polyacrylic acid,
10 polymaleic acid, and polyacrylic/polymaleic and copolymers and their salts, such as those sold by BASF under the trade mark Sokalan.

The builders generally constitute from 0 to 3 wt%,
15 more preferably from 0.1 to 1 wt%, by weight of the compositions.

Compositions which comprise an enzyme may optionally contain materials which maintain the stability of the
20 enzyme. Such enzyme stabilizers include, for example, polyols such as propylene glycol, boric acid and borax. Combinations of these enzyme stabilizers may also be employed. If utilized, the enzyme stabilizers generally constitute from 0.1 to 1 wt% of the compositions.

25 The compositions may optionally comprise materials which serve as phase stabilizers and/or co-solvents. Example are C₁-C₃ alcohols or diols such as methanol, ethanol, propanol and 1,2-propanediol. C₁-C₃
30 alkanolamines such as mono-, di- and triethanolamines and monoisopropanolamine can also be used, by themselves or in combination with the alcohols.

If the composition is in liquid form, it may be anhydrous, or, for example, contain up to 5 wt% water. Aqueous compositions generally contain greater than 8 wt% 5 water based on the weight of the aqueous composition. Desirably the aqueous compositions contain more than 10 wt%, 15 wt%, 20 wt%, 25 wt% or 30 wt% water, but desirably less than 80 wt% water, more desirably less than 70 wt%, 60 wt%, 50 wt% or 40 wt% water. They may, 10 for example, contain from 30 to 65 wt% water.

The compositions may optionally comprise components which adjust or maintain the pH of the compositions at optimum levels. Examples of pH adjusting agents are NaOH 15 and citric acid. The pH may be from, for example, 1 to 13, such as 8 to 11 depending on the nature of the composition. For example, a dishwashing composition desirably has a pH of 8 to 11, a laundry composition desirably has a pH of 7 to 9, and a water-softening 20 composition desirably has a pH of 7 to 9.

The composition, such as a washing composition within the container, capsule or receptacle part, or within a compartment thereof if there is more than one 25 compartment, need not be uniform. For example during manufacture it could be fed first with a settable agent, for example a gel, useful in a washing process, and then with a different material. The first material could dissolve slowly in the washing process so as to deliver 30 its charge over a long period within the washing process. This might be useful, for example, to provide delayed or

sustained delivery of a softening agent in a clothes washing capsule.

The composition, such as a washing composition may, especially for dishwashing or laundry, include a tablet. Preferably a tablet contains a material useful in a washing process and is formulated to provide slow release of that material during a washing process and/or delayed release thereof. Delayed release may be achieved by providing the tablet with a coating which is slow to dissolve during the washing process. Alternatively the tablet may provide a quick release of components required early in the wash, for example water-softening components and/or enzymes. The tablet may, for example, comprise a disrupting agent, such as one which effervesces when in contact with water such as a combination of citric acid and an alkali metal carbonate or bicarbonate.

A tablet may be provided in the main volume of the receptacle part or may be provided in an outwardly facing opening or depression, as previously described.

When a washing capsule of the invention has a tablet retained in an outwardly facing opening or depression the tablet is preferably one which will not transfer any washing composition to the hands of a user. For example, it may be coated with a soluble polymeric material. As mentioned above, this may also be desirable for delayed release of its charge. If it is desired that the tablet dissolves quickly it may, for example, comprise a disrupting agent such as an effervescent agent.

In accordance with a further aspect of the invention there is provided a method of ware washing, comprising use of a container, receptacle or washing capsule as described and defined above, the method entailing

5 introducing the container, receptacle or washing capsule into a ware washing machine such as a laundry washing machine or dishwashing machine, prior to commencement of the washing process, the container, receptacle or washing capsule being entirely consumed during the washing

10 process.

The invention also provides a capsule - that is to say, a container for the relevant ingredients, which container is in at least two parts (a body part and a cap part) which fit tightly, and preferably sealingly and inseparably, together to form a compartment in which is stored the ingredient to be delivered. In one example - see Figure 11A in the accompanying Drawings - the capsule may have a body and cap each provided with a central

15 axially-parallel partition, so that the capsule as a whole has two separate compartments. In another example the capsule may have three parts - a body, a first cap, and then a second cap to fit over the closed end of either the body or the first cap, so as again to result

20 in a capsule with two separate compartments. And where there are two or three such parts (or more; four parts - a body and three caps - make three compartments, and so on), then naturally the ingredients in each compartment may be the same or they may be different.

25

30

The capsule of the invention is one that dissolves in the destined aqueous medium to release its contents

therein. The term "dissolve" is used herein in a fairly general sense, to indicate that the capsule crumbles, decomposes, disintegrates or disperses; it need not actually dissolve, although in most cases it will.

5

The invention will now be further described, by way of example, with reference to the accompanying drawings in which.

10 Fig. 1 is a perspective view, generally from above, of an array of receptacle parts;

Fig. 2 is a perspective view, generally from above, of an alternative array of receptacle parts;

15

Fig. 3 is a perspective view of some of the parts shown in Fig. 2, but looking generally from underneath;

20 Fig. 4 is a perspective view, generally from above, of a third embodiment of receptacle part;

25 Fig. 5 is a perspective view, generally from above, of the Fig. 4 embodiment, but filled with washing composition and closed over by a closure part, to form a washing capsule of the invention;

Fig. 6 is a perspective view from above of a fourth embodiment of receptacle part; and

30 Fig. 7 is a perspective view from below of receptacle parts of the type shown in Fig. 6.

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Fig. 8A & B show longitudinal cross-sections of a capsular container of the invention in its open and closed states respectively;

5 Fig. 9 shows the closed capsular container of Fig. 8B but in see-through perspective;

10 Fig. 10A & B show longitudinal cross-sections of two- and three-compartment capsular containers of the invention;

15 Fig. 11A & B show respectively longitudinal and transverse cross-sections of another two-compartment capsular container of the invention;

Fig. 12 shows a section through the wall of a solid-filled polymer capsule of the invention;

20 Fig. 13A-M show various different forms of moulding on and in the surface of capsular containers of the invention.

25 Fig. 1 shows an array of eight receptacle parts 2, arranged as two columns and four rows. Each receptacle part has a flat base wall without indentations or recesses and four uprights side walls 4, and has no top wall. Thus, each receptacle part is upwardly open. Around its opening, at the top of the side walls 4, is an outwardly-directed flange 6, which extends around the 30 entire opening. The receptacle parts are joined to adjacent receptacle parts by webs 8 between the flanges 6. The flanges 6 of all of the receptacle parts lie in

one plane. The base walls of all of the receptacle parts also lie in one place, parallel to the plane in which the flanges lie.

5 The array shown in the drawing is made by injection moulding. The thermoplastic polymer employed in this embodiment is polyvinyl alcohol, and is translucent. The wall thickness is about 0.7 mm. The resulting moulded array is self-supporting.

10 After injection moulding score lines may be cut into the webs 8 between the flanges, to aid the breaking apart of the washing capsules, for use.

15 The moulded array is fed to a filling zone where the receptacle parts are simultaneously filled via eight nozzles, with a dishwashing composition. The dishwashing composition could be a powder, gel or paste or could be a liquid formulation. If it is a liquid it may be a liquid 20 formulation of relatively low water content, for example, 2 to 5 wt%, given the properties of the polymer. Alternatively the water content may be higher, for example up to 60 wt% or even 80 wt%, so long as the PVOH is not attacked by the composition. Such steps are 25 described above. A translucent cover film is then laid over the array and heat sealed against the flanges 6, so that each receptacle part has, over it, a closure part. The closure part is also of polyvinyl alcohol, but is much thinner, about 80 μm in this embodiment.

30 Although the film which constitutes the closure parts is tough it will be appreciated that it is

generally less robust than the receptacle parts. In this case, before packaging the product, the capsules may be put into face-to-face contact. An array of washing capsules identical to that of the drawing may be placed 5 in face-to-face contact with it. Alternatively, and conveniently, the array shown in the drawing may be folded about line A-A shown in Fig. 1.

The drawing illustrates the invention but in 10 practice an array of receptacle parts is likely to be considerably larger. Nevertheless, the manufacturing method would be as described.

In use, a user will simply break off a washing 15 capsule from the array, and put it in the dishwashing machine. During the washing process the entire washing capsule will dissolve. The first part to dissolve will generally be the closure part. This may happen very quickly once the washing process starts and the washing 20 composition will immediately be delivered. The receptacle part will generally dissolve more slowly but it will have dissolved entirely by the end of the washing process.

25 Figs. 2 and 3 show an alternative embodiment of the receptacle parts. The receptacle parts shown in Figs. 2 and 3 are of similar shape and size to those shown in Fig. 1, but have, within the main chamber defined by the base wall and side walls of each receptacle part, a 30 generally cylindrical upstand 10, in a central position. Each upstand is open at its upper end, and its upper end is in the same plane as the flange 6.

As shown in Fig. 3, each receptacle part also has a depression 12 at a central position in its base wall. The depression is relatively shallow, and it is aligned 5 with the upstand 10 carried by the base wall on its other side. Each depression contains within it a tablet 14. Each tablet contains a washing composition or a material which forms part of a washing composition, but is formulated for quick release, slow release and/or delayed 10 release. For slow release it may be a tablet which dissolves over an extended period. For delayed release it may be a tablet coated with a polymeric coating which is slow to dissolve, so that it releases its charge in the middle or towards the end of a washing cycle.

15

Another difference between the embodiment of Fig. 2 and that of Fig. 1 is that in the Fig. 2 embodiment there is a plurality of breakable webs 16 of polymeric material extending between the flanges of adjacent receptacle 20 parts.

The array shown in Figs. 2 and 3 is again made by injection moulding, using HPMC polymer having a wall thickness of about 0.8 mm, although PVOH, for example, 25 may also be used. Tablets 14 are press-fitted into the depressions 12 in the undersides of the base walls. The array is then inverted for filling. The upstands 10 are filled with one material, and the remaining volumes, between the upstands and the side walls of the respective 30 receptacle parts, are filled with another material. A cover film is then laid over the array and heat sealed against the flanges 6 and against the ends of the

upstands 10, so that each receptacle part has, over it, a closure part. The closure part is of HPMC, about 70 microns thick. Again, PVOH may, for example, also be used.

5

The embodiment shown in Figs. 4 and 5 is similar to that of Figs. 2 and 3 in having an upstand. However the remaining volume of the receptacle part is divided into two by means of walls 18, 20, extending from the upstand 10 in opposed directions, and with each connecting with a respective side wall of the receptacle part. It will be apparent that the receptacle part comprises three main chambers whose contents are released into the washing water once the closure part dissolves. One chamber 22 is 15 defined within the upstand and the other chambers 24, 26 are of identical size to each other and are defined between the upstand and the side walls. The underside of the receptacle part may, like the embodiment of Figs. 2 and 3, comprise a central depression into which is 20 pressed a tablet. The receptacle parts are formed, in an array, by injection moulding.

Fig. 5 shows a washing capsule which uses the receptacle part shown in Fig. 4. The receptacle part has 25 been filled with three different materials useful in a dishwashing cycle and a cover film is shown in place.

The embodiment of Figs. 6 and 7 is simpler than those of Figs. 2 to 5. The receptacle part shown does 30 not have a central upstand. There is one main volume. However the underside of the base wall is moulded with a depression and into this depression is press-fitted a

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tablet. In the embodiment of Figs. 6 and 7 the main chamber of the receptacle part can be filled with two or more gels which stay separate, for example, side by side, or one within the other, or in the form of separate 5 stripes. The receptacle parts of Figs. 6 and 7 may be formed, in an array, by vacuum forming.

In the embodiments of Figs. 4 to 7 the materials selected for the receptacle parts and closure parts, and 10 their thicknesses, are as described for the Fig. 1 embodiment.

Figure 8 shows a two-part, one compartment capsular container of the invention in its open and its closed 15 form.

The body (111) and cap (112) are to be welded together and are made so that the open end (111a) of one will pass into the open end (112a) of the other with the 20 smallest gap that can be practically achieved to allow easy assembly. There is a "stop" - a ridge (111b) running all round outside of the body 111 that co- operates with a groove (112b) running all round the inside of the cap 112 - so that the entry of one into the 25 other cannot overrun, and stops at the same fixed position in every case.

When the two halves or shells 111, 112 are in the closed position (as in Figure 8B), with the entire 30 periphery of the open end 111a of the body 111 overlapped by the periphery of the open end 112a of the cap 112, the capsular container is ready for welding. The welding

- 66 -

equipment (not shown) forms a weld line (113) between the two layers all round the periphery of the container.

Figures 10 and 11 show different sorts of multi-compartment capsular container according to the invention.

In Figure 10 the container is made in two or more parts (three in Figure 10A, four are shown in Figure 10B, but there could be more) - in each case there is a single cap portion (132) and a plurality of body portions (as 131). The outer of the body portions 131 is much the same as an "ordinary" body portion (as in Figure 8), but each inner one is shaped at its "outer" end (131c) so that it will fit tightly inside the open mouth of the next body portion, much like in Figure 8 the body 111 fits inside the cap 112.

As shown (in Figure 10A), when the first (outer) body part 131 has been filled with product A, it may then be closed by the second (inner) body part 131 within it. That second body part 131 may then be filled with product B, the cap 132 placed in position, and the three parts welded together at the same time.

25

Figure 11 shows a capsular container with body (141) and cap (142) two compartments side-by-side (Figure 11B shows a transverse section on the line A-A in Figure 11A). The two compartments can of course hold different products (A and B).

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There is theoretically no limit to the number of separate chambers that can be produced either linearly (as in Figure 10) or side by side within the body portion (as in Figure 11). Of course, limitations will be set by 5 practical problems of manufacture.

In Figure 12 there is shown a section through the wall of a solid-filled polymer capsular container of the invention.

10

Inert solids in powder form have been added to the polymer formulation prior to moulding. This provides a more rigid shell. It especially provides a more rigid capsule shell with a surface less immediately affected by 15 the aqueous content of the mouth or oesophagus, thereby reducing surface tackiness during the initial swallowing. The capsule surface is to a significant extent made up of the particulate insoluble solid ingredient (as 154); the soluble polymer (155) is partially concealed below the 20 contact surface (156).

Figure 13 etc show various different forms of moulding on the surface of capsular containers of the invention, some in the form of cross-sections.

25

These are self-evident, and need little comment. Figure 13A, F, for example, shows a capsular container with longitudinal raised ribs, while Figure 13B shows one with lateral (or circumferential) raised ribs and Figure 30 13E shows one with helical ribs. Figure 13C, H shows a container with raised pimples, while Figure 13D, I shows one with raised identification coding patterns. Figures

13G, J, K, L and M show variants analogues to some of the others, but with incuse rather than raised portions.

The invention is further explained in the following
5 Examples.

EXAMPLES

Example 1:

10 The manufacture of capsules by injection moulding and
laser welding

The moulding stage

15 Capsules according to the invention were made by the injection moulding method utilising an Arborg 220D (35 tonne) injection moulding machine. The injection cavities were in a two-impression (cap/body) composite water-cooled stainless-steel mould. The PVOH had a
20 material melt flow index of 10-20 grams per 10 mins (DIN 53735).

25 Injection temperatures were 175°C, 180°C, 180°C and 185°C in the feed, zone 2 and 3, and Nozzle areas. The first stage injection pressure was 400psi (.....), and the hold stage pressure was 270psi (.....). The pressure well time was 3 secs in the first stage and 5 secs in the hold stage. Tool temperatures were between ambient and 40°C.

30

The moulding pressures were just sufficient to fill the cavities on the first pressure stage and then

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sufficient packing pressure to hold on the second stage. Mould open and close rates were as fast as possible.

As noted, the mould layout was divided into two
5 halves, one half moulding capsule bases and the other
half capsule caps. After the mould opening sequence, two
robotically controlled loading plates pneumatically
picked up each capsule half from each tool face. With
identical cavity pitch centres, these loading plates were
10 brought together so that each capsule half was located
resulting in the usual temporary location of the pair
ready for automatic filling.

The filling stage

15

For test purposes the capsules were filled by hand
with various test materials (see below).

The welding stage

20

The closed capsule is introduced into a transparent
tube with an internal diameter not more than 20% greater
than the external diameter of the capsule. An array of
diodes is located circumferentially around the outside of
25 the tube. As the capsule passes by the diode array, a
weld is formed. The velocity of the capsule and the
power of the IR emitted by the diode array provide the
necessary control over the melting process. The IR
emission is either continuous or discontinuous. In the
30 case of discontinuous emission, this is achieved by
synchronisation of switching depending on the form of

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weld required and the sensitivity of the contents of the capsule to the IR.

If the characteristics of the material contained
5 within the capsule are such that they absorb the IR,
switching of the laser is necessary such that exposure to
the IR is limited to the area of the join. This is
effected by means of electrical switching or, in a
further embodiment, by a form of optical switching using
10 a lens/prism arrangement. In order to overcome the
difficulty of synchronisation, again optical fibre
delivery of the IR is used to restrict the area of
exposure.

15 Example 2:

The manufacture of capsules using laser welding

In an alternative laser welding stage, the laser or
other IR source is arranged to focus on the area of the
20 join. This does not create a full circumferential weld
but generates a spot weld. Again, the laser is
continuously emitting. By forcing the filled capsules to
roll (by mechanical means) whilst exposed to the laser, a
full circumferential weld results. Alternatively, an
25 optical fibre is used to deliver the IR to the join.

Test Results

PVOH capsules made in the manner described in
30 Example 1 above were filled with either sugar or
tea leaves. They were designed to have a cap portion

that would dissolve sooner than the body, and thus open the capsule progressively.

Similarly, a number of conventional gelatine 5 capsules were also prepared and so filled.

In the Test, a capsule was placed in each Test Subject's mouth (in the buccal cavity), and the Subject was asked to note when he/she became aware of the taste 10 of the contents - thus, when the capsule "opened" - and then when the capsule had completely dissolved.

There were two Test Subjects, and each Test was carried out twelve times (for each filling).

15 The conventional gelatine capsules opened in 3-4 minutes, and dissolved completely in 5-8 minutes. The sugar-filled PVOH capsules of the invention opened in 8-12 minutes, while the tea-filled ones took longer - 20 14-18 minutes. Complete dissolution took 30-40 minutes in each case.

CLAIMS

1. A rigid, water-soluble container made of an injection moulded poly(vinyl alcohol) and/or a cellulose ether, which container encases a fabric care, surface care or dishwashing composition.
2. A container according to claim 1 which encases a dishwashing, water-softening, laundry, detergent, rinse aid, disinfectant, antibacterial or antiseptic composition or a refill composition for a trigger-type spray.
3. A container according to claim 1 or 2 which has two or more compartments.
4. A container according to any one of the preceding claims wherein the composition has a mass of at least 15g.
5. A container according to claim 4 wherein the composition has a mass of from 15 to 40g.
6. A container according to any one of the preceding claims which comprises a poly(vinyl alcohol).
7. A container according to any one of the preceding claims which comprises hydroxypropylmethylcellulose.
8. A container according to any one of the preceding claims wherein the container is sealed by a water-soluble

closure part in the form of a film or injection-moulded, rigid closure.

9. A container according to claim 8 wherein the closure 5 part comprises a poly(vinyl alcohol) film or closure.

10. A container according to any one of the preceding claims which contains a dishwashing, water-softening, laundry or detergent composition or a rinse aid.

10

11. A container according to any one of claims 1 to 9 which contains a disinfectant, antibacterial or antiseptic composition.

15

12. A container according to any one of claim 1 to 9 which contains a refill composition for a trigger-type spray.

20

13. A capsule comprising a self-supporting receptacle part and a closure part, the receptacle part and the closure part together enclosing a fabric care, surface care or dishwashing composition, the receptacle part being formed of a water-soluble polymer, and the closure part being formed of a water-soluble polymer, wherein, in 25 use, the closure part dissolves before the receptacle part.

30

14. A capsule according to claim 13 wherein the water-soluble polymer is a poly(vinyl alcohol) or a cellulose ether.

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15. A capsule comprising a self-supporting receptacle part and a closure part, the receptacle part and the closure part together enclosing a fabric care, surface care or dishwashing composition, the receptacle part
5 being formed of a water-soluble polymer, and the closure part being formed of a water-soluble polymer, wherein the water-soluble polymer is a poly(vinyl alcohol) or a cellulose ether.

10 16. A capsule according to claim 15 wherein, in use, the closure part dissolves before the receptacle part.

15 17. A capsule according to any one of claim 13 to 16 which is a washing capsule enclosing a washing composition.

20 18. A capsule according to any one of claims 13 to 17 wherein the receptacle part has side walls which terminate at their upper end in an outward flange, to which the closure part is sealingly secured.

25 19. A capsule according to any one of claims 13 to 18 wherein the closure part is a plastics film.

30 20. A capsule according to any one of claims 13 to 19 wherein the composition comprises a powder, gel, paste or low water liquid formulation.

21. A washing capsule according to any one of claims 13 to 20 wherein the washing capsule comprises a tablet formulated for delayed and/or sustained release of a material.

22. A capsule according to any one of claims 13 to 21 wherein the receptacle part defines two or more compartments which contain different products.

5

23. A capsule according to claim 22 wherein the receptacle part comprises an upstanding wall which separates compartments thereof.

10 24. A capsule according to any one of claims 13 to 23 wherein the receptacle part comprises an outwardly facing opening into which a tablet, preferably comprising a material useful in a washing process, is press-fitted.

15 25. A capsule according to any one of claims 13 to 24 where the washing composition contains an enzyme.

20 26. A capsule according to any one of claims 13 to 25 wherein the closure part is a transparent or translucent material.

27. A washing capsule according to any one of claims 13 to 26 wherein the water-soluble polymer is a poly(vinyl alcohol).

25

28. An array of washing capsules as defined in any one of claims 13 to 27 which are joined together but are readily separable from each other for use.

30 29. An array according to claim 28 wherein the array has a line of symmetry extending between capsules, and the two halves of the array are folded together about the

line of symmetry, with the closure parts in face-to-face contact.

30. A method of manufacturing an array of washing
5 capsules as defined in claim 28 or 29, which method
comprises: forming an array of receptacle parts, each
receptacle part being connected to adjacent receptacle
parts but being separable from them by a snap or tear
action; charging the receptacle parts with washing
10 composition; and sealingly securing a sheet of a water-
soluble polymer over the top of the array, to form the
closure parts for all the receptacle parts of the array.

31. An injection-moulded capsule container of any size
15 or shape for the delivery of a water-destined ingredient
selected from a fabric care, surface care or dishwashing
composition, which container is made of a material that
will dissolve in the intended aqueous destination site.

20 32. A container according to claim 31 wherein the
composition is a detergent, biocide, deodorant or water-
treatment chemical.

33. A container according to claim 31 or 32 wherein the
25 material that will dissolve is a poly(vinyl alcohol) or a
cellulose ether.

34. A container according to any one of claims 31 to 33
which is in the form of an elongate tubular package with
30 closed, rounded ends.

35. A method of ware washing, comprising use of a container, receptacle or washing capsule as defined in any one of the preceding claims, the method entailing introducing the container, receptacle or washing capsule 5 into a ware washing machine prior to commencement of the washing process, the container or washing capsule being entirely consumed during the washing process.

36. A capsule container comprising at least two 10 components made of one or more material(s) that can be moulded and which are water soluble or water dispersible or in which a substantial part of the surface of these components is water soluble or water dispersible so as to leave perforations throughout the wall when the capsular 15 container is placed in contact with an aqueous environment, wherein the container has one to six compartments, preferably one or two or three, the content of the various compartments being accessible to the aqueous environment when the capsular container is exposed to such an aqueous environment, the accessibility 20 time of the various compartments being the same or different from one compartment to another compartment, with the proviso that the content of the container is not a fabric care, surface care or dishwashing composition.

25

37. Container according to claim 36 wherein the moulded compartments are made from injection moulding material.

38. Container according to claim 36 wherein the moulded 30 components are wholly water soluble or water dispersible.

39. Container according to any of claims 36 to 38 wherein the material(s) are soluble in an aqueous environment at 5°C or higher.

5 40. Container according to any of claims 36 to 39 wherein the material(s) are soluble in an aqueous environment between 35°C and 37°C.

10 41. Container according to any of claims 36 to 40 with at least two compartments, preferably two or three.

15 42. Container according to any of claims 36 to 41 wherein the accessibility time of the various compartments to the aqueous environment is different from one compartment to another compartment.

43. Container according to any of claims 36 to 42 wherein the components are one body and one or more caps, preferably one or two caps.

20 44. Container according to any of claims 36 to 43 wherein the difference of accessibility time to an aqueous environment from one compartment to another one is in the range of 1 minute to 12 hours at the same 25 temperature in the range of 5°C to 95°C.

45. Container according to any of claims 36 to 44 wherein the container is made from one or more materials which are water-soluble polymers.

30 46. Container according to claim 45 where the materials is polyvinyl alcohol or cellulose derivatives.

47. Container according to claim 46 where the material is polyvinyl alcohol.

5 48. Container according to any of claims 36 to 47 wherein the container contains at least one active ingredient in each compartment, and where if more than one compartment, the ingredients are different.

10 49. Container according to any of claims 36 to 48 wherein the closing of the capsular container by putting the cap on the body has the effect of separating the compartments from each other.

15 50. Container according to any of claims 36 to 49 wherein the closing of a compartment by putting a cap on a body allows that compartment to be separated from the next one.

20 51. Container according to any of claims 36 to 50 wherein the components are welded to form a single indivisible unit.

25 52. Container according to claim 51 wherein the welding is on a line around the container where preferably the line is situated on a planar cross-section of the container.

30 53. Container according to claim 51 or 52 wherein the welding is effected by laser welding.

54. Container according to claim 53 wherein either

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- (a) one of the surfaces before welding is coated with a laser beam reflecting ingredient, or
- (b) one of the components is moulded with a laser beam reflecting ingredient contained within it.

5

55. Container according to any of claims 36 to 54 wherein the accessibility time of the various compartments is due to the difference in thickness of the wall of the compartment.

10

56. Container according to claims 36 to 55 wherein the thinner area(s) are made of a water soluble or water dispersible coating covering perforation(s) in the wall of any component.

15

57. Container according to any of claims 36 to 54 wherein the accessibility time of the various compartments is due to differences in the nature of the polymers used to make different compartments.

20

58. Container according to any of claims 36 to 57 wherein a component has a conical shape.

25

59. Container according to claim 55 wherein the thinner areas of the walls are disposed longitudinally according to the general elongated shape of the capsular container.

30

60. A capsular container according to any of claims 36 to 59 having raised portions on its external surface and wherein the raised portions are in the form of short, small pimple like projections or ribs that extend wholly or partially either around or along the capsule, and/or

of markings allowing identification of the capsular contained and/or its contents.

61. A capsular container according to any of claims 36
5 to 60 having raised portions on its external surface and wherein the raised portions provide an incuse pattern design on a capsular container of standard form but with relatively thick walls, so forming an array of thin-walled panels such that in use the thin-walled panels
10 quickly dissolve, leaving the capsule with a grid structure of holes.

62. Container according to any of claims 36 to 61 wherein the wall of the container contains particles
15 which are susceptible to accelerate the rate of dissolution of the capsular container.

63. Container according to claims 62 wherein the particles are made of a material susceptible to react
20 chemically with the environment in which the capsular container is to be used, the reaction causing preferably an effervescence.

64. Container according to claim 62 or 63 where the
25 particle size of is 1 to 100 microns, preferably from 2 to 80 microns.

65. Container according to any of claims 62 to 64 wherein the material of the particle is selected in the
30 group comprising sodium or potassium or magnesium carbonate or bicarbonate; tartaric acid, citric acid, cinnamic acid; or the salts thereof.

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66. A capsular container according to any of Claims 36 to 65 for a pharmaceutical or nutraceutical use or when used for a pharmaceutical or nutraceutical purpose.

5

67. Container according to claim 66 for delivery of one or more pharmaceutically or nutraceutically active ingredients into a human or animal body.

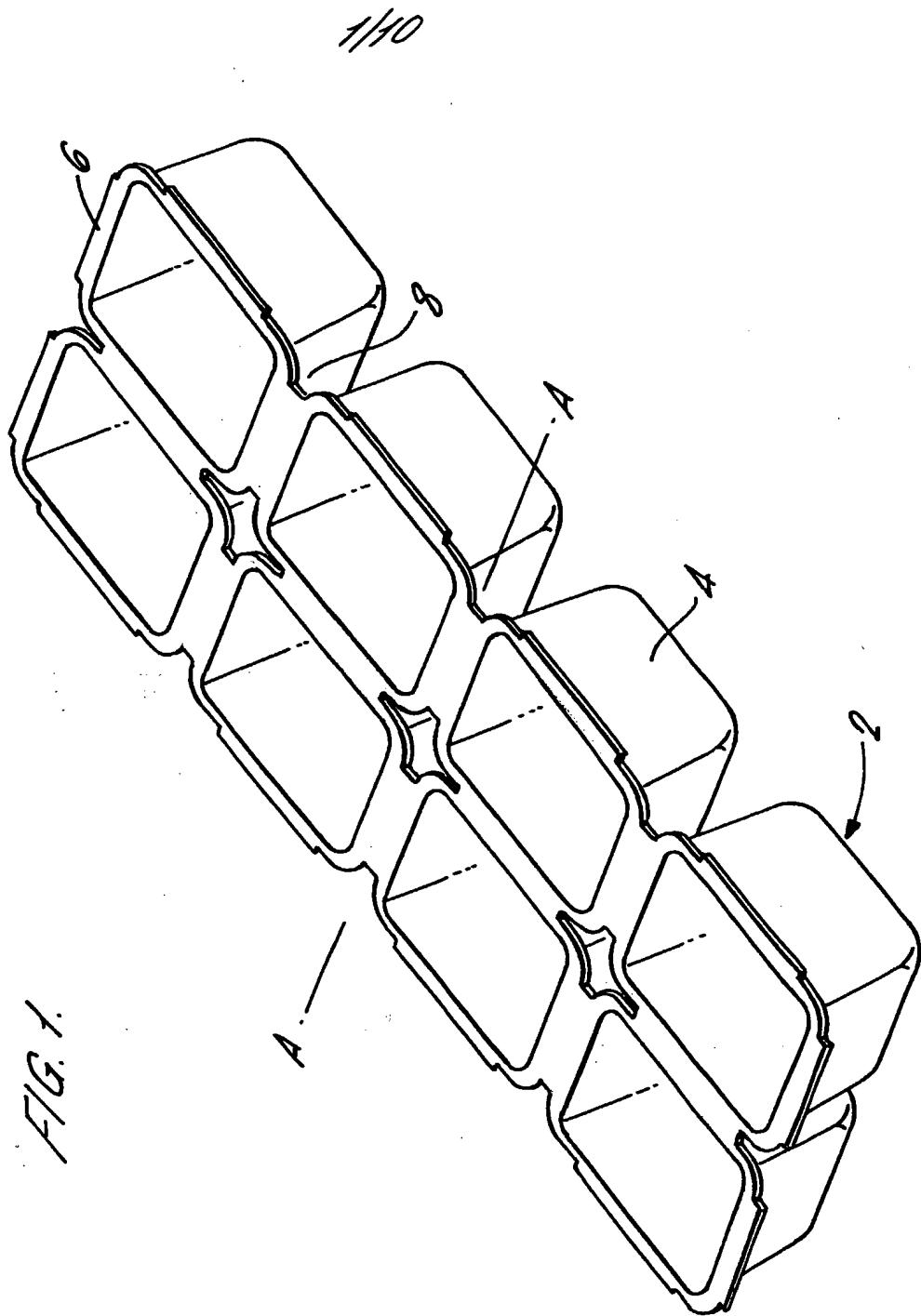
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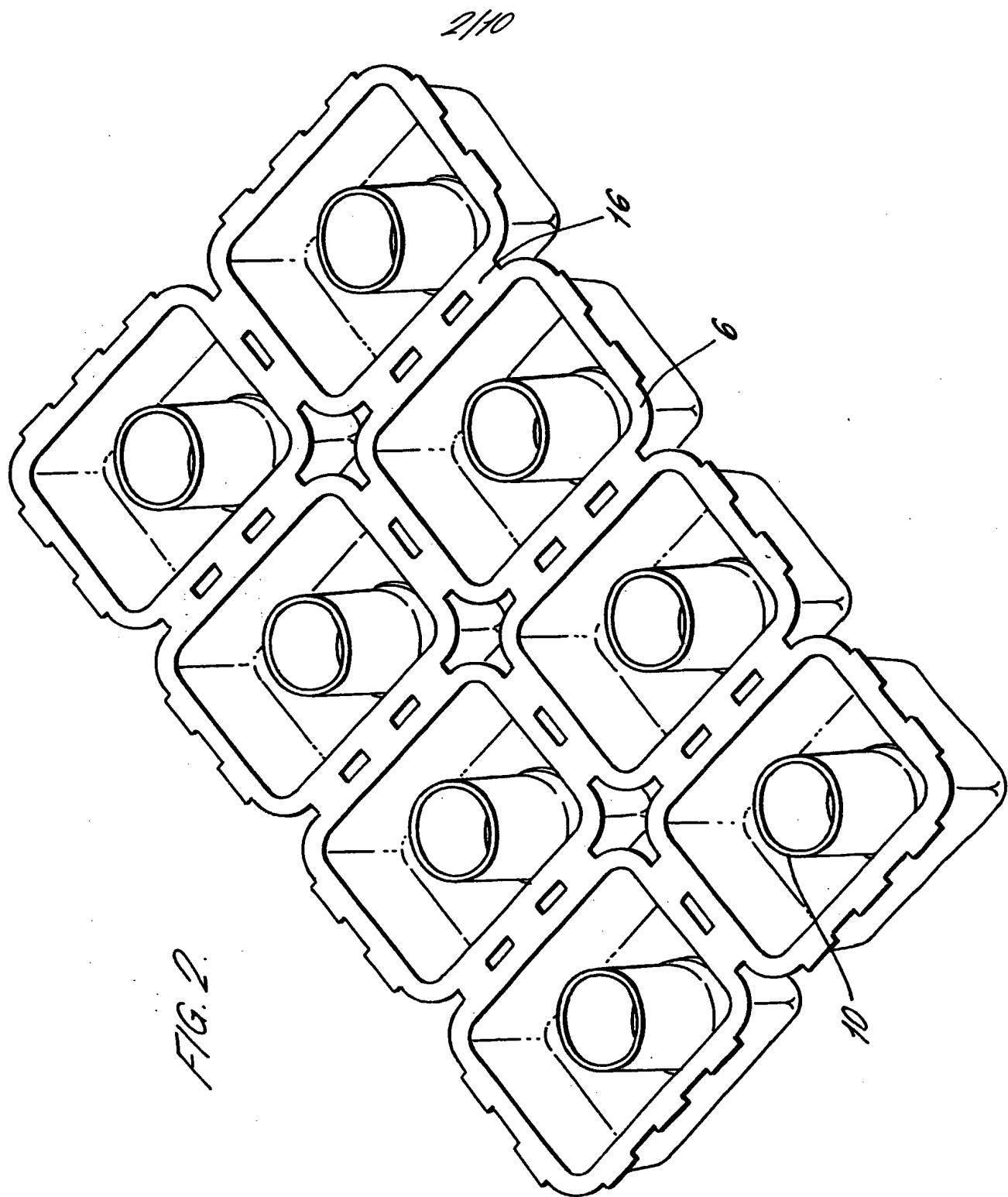
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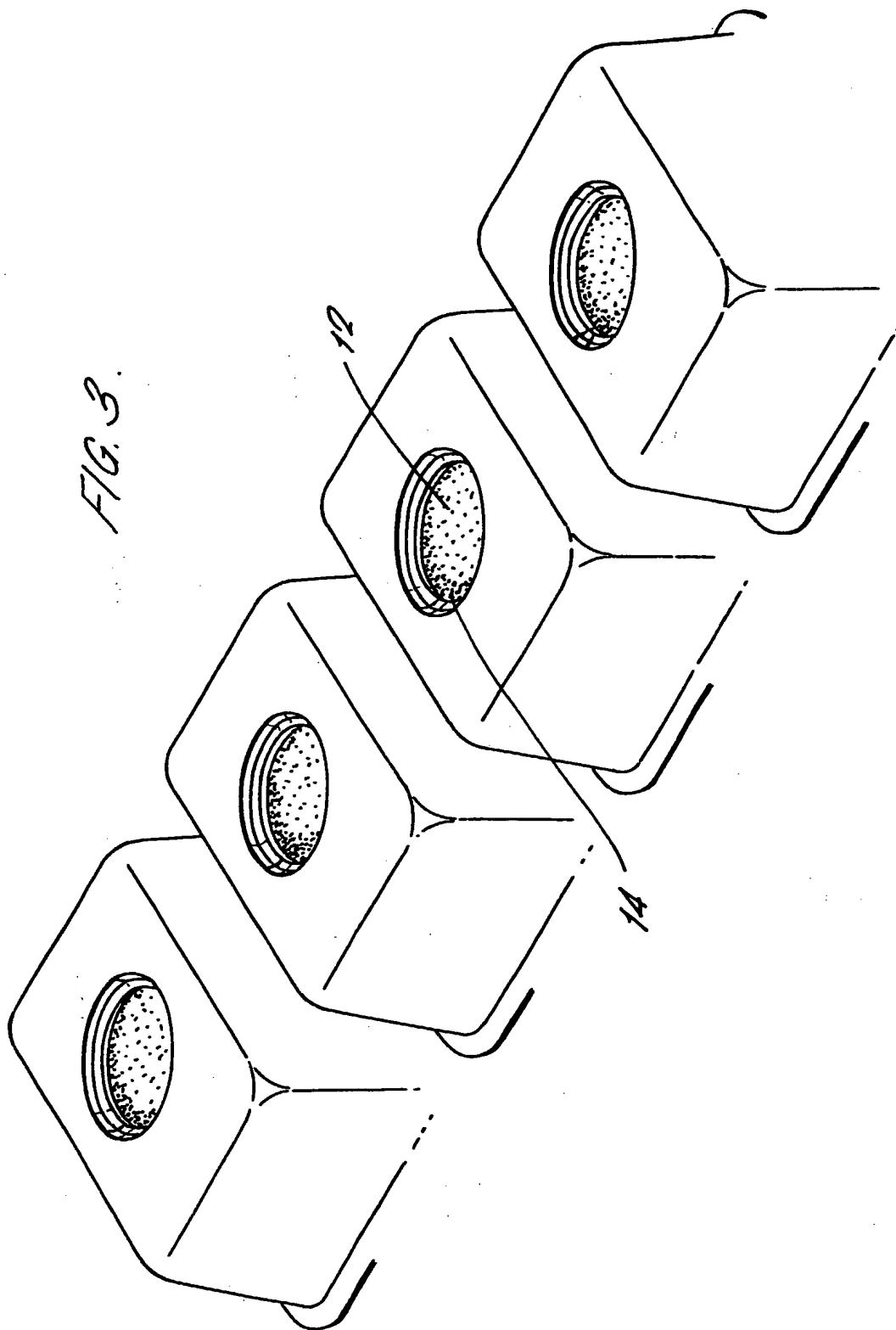
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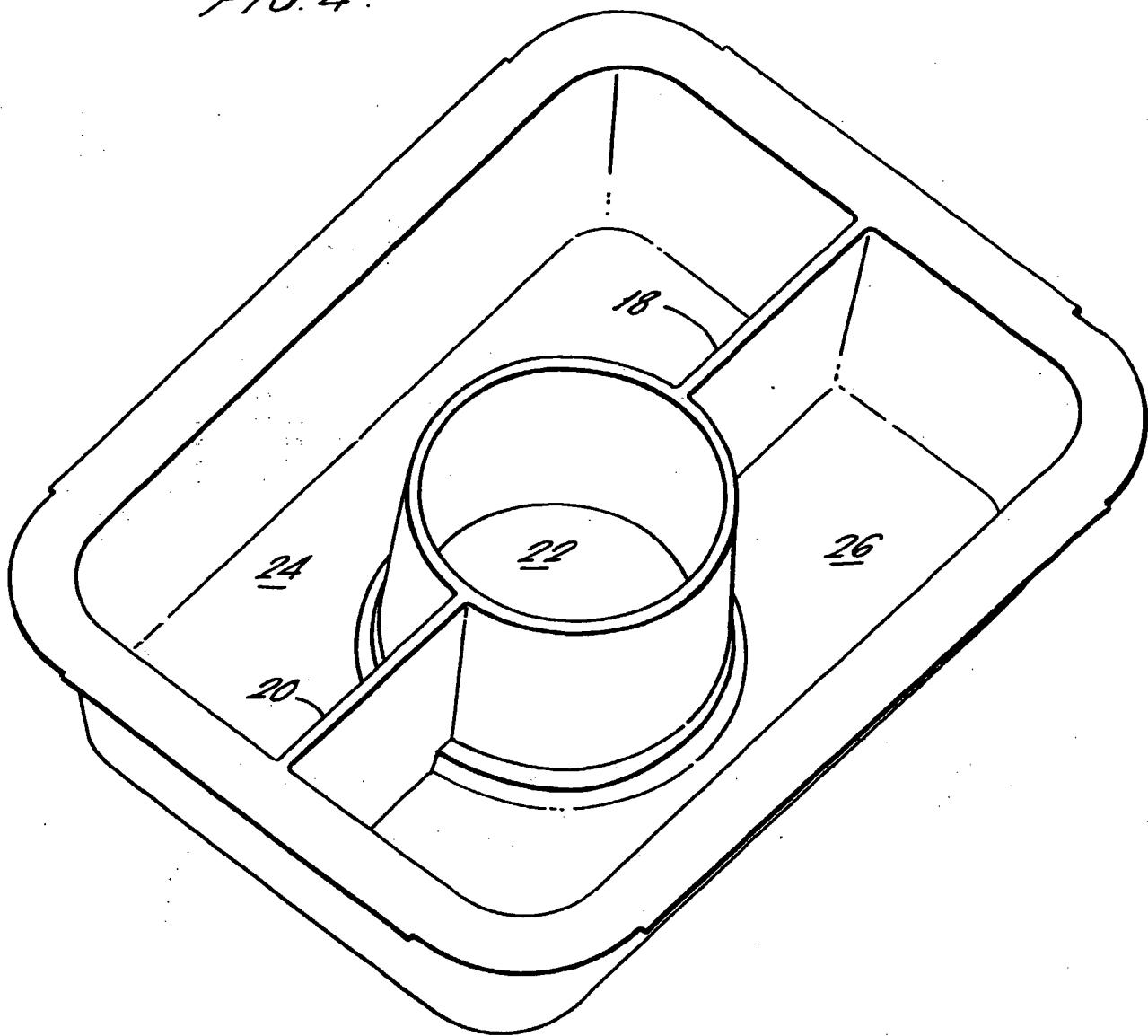
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FIG. 3.

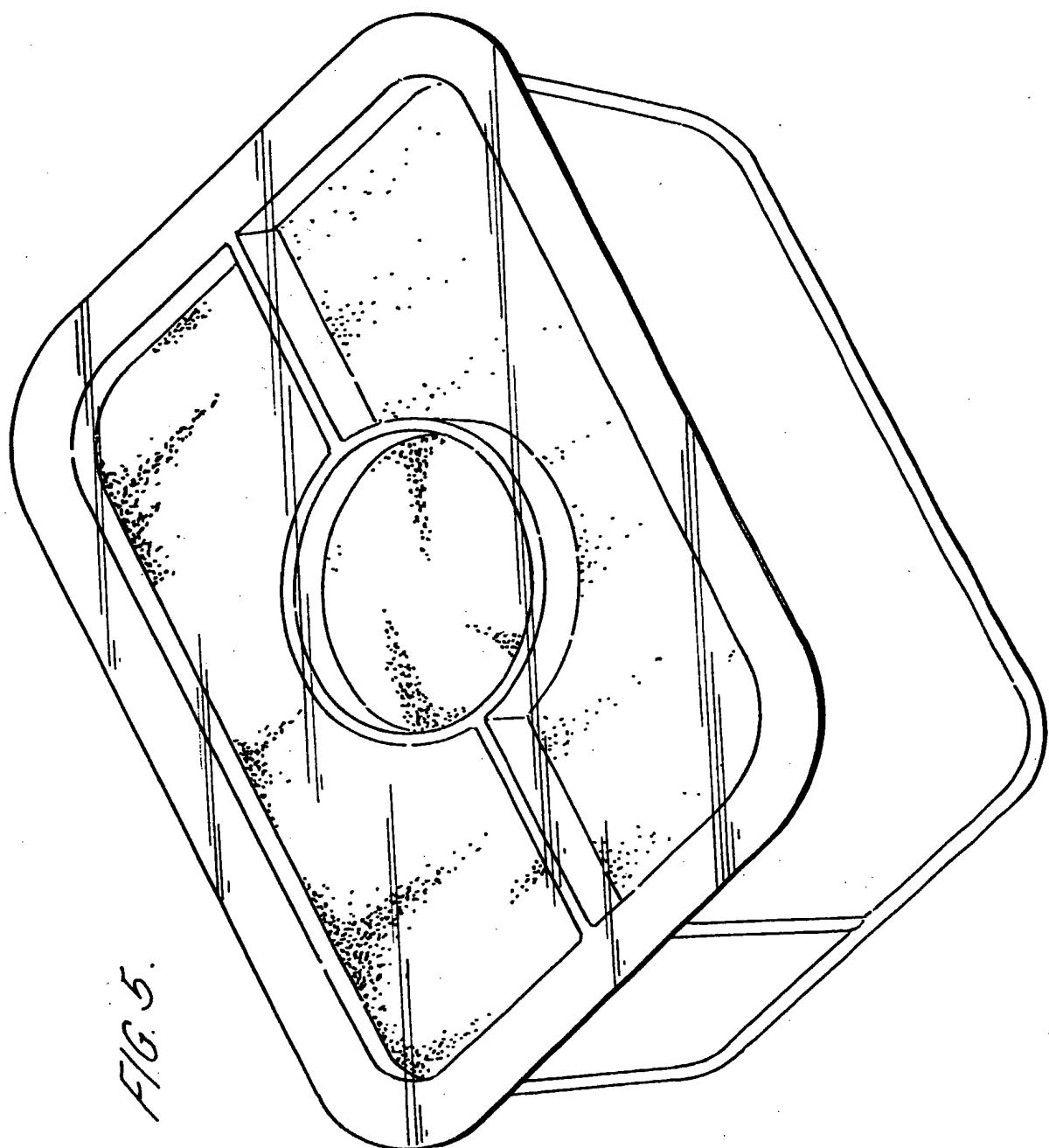


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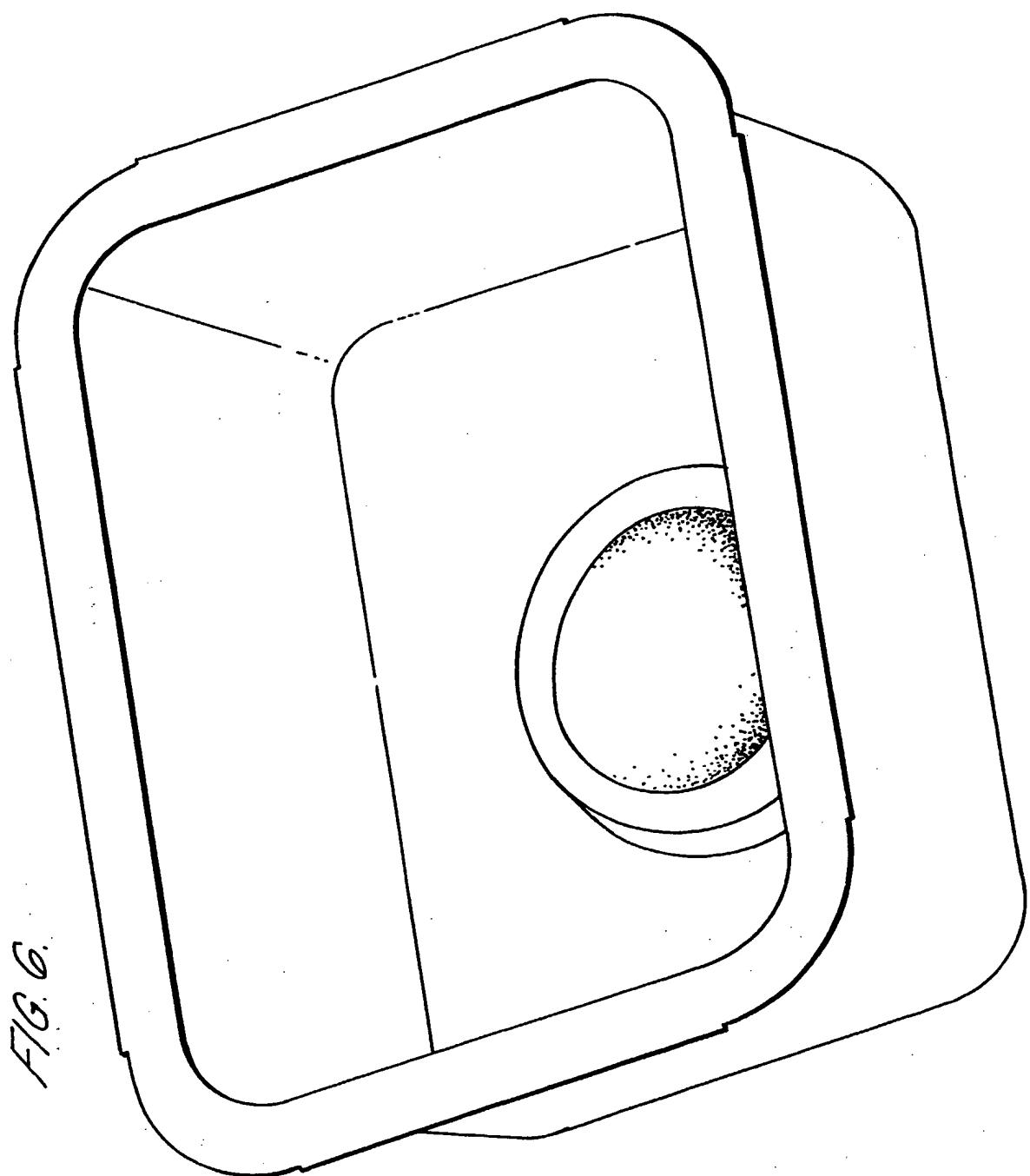
FIG. 4.

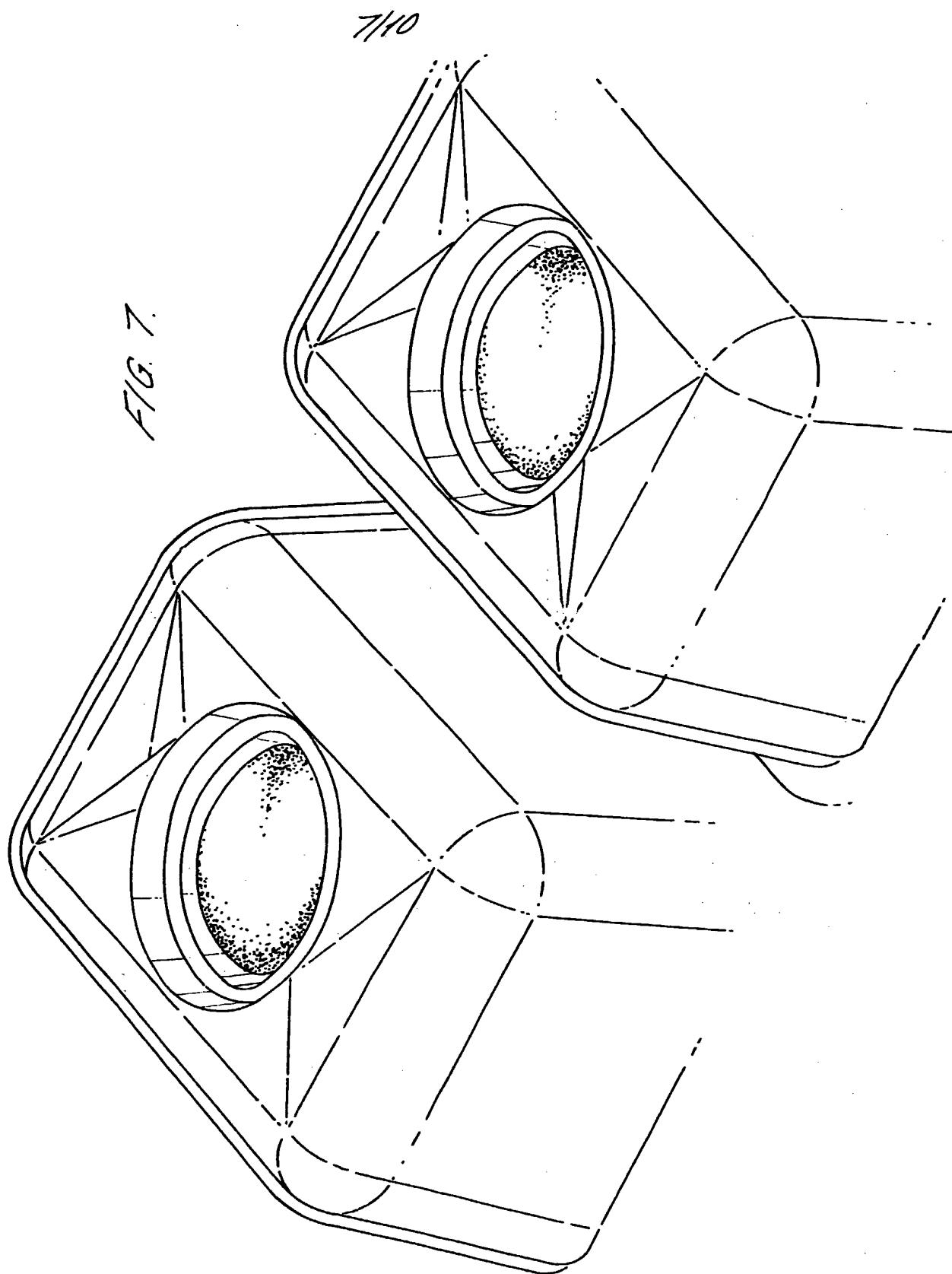


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FIG. 8A.

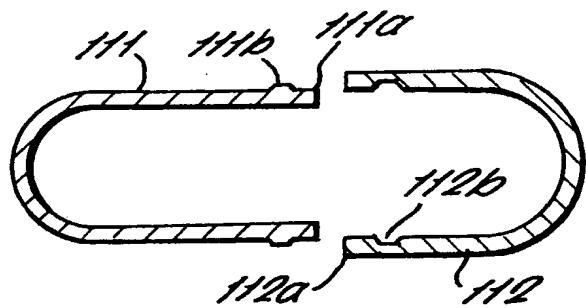


FIG. 8B.

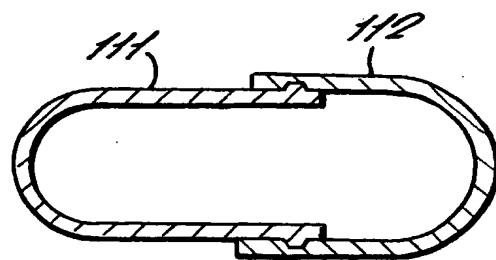


FIG. 9.

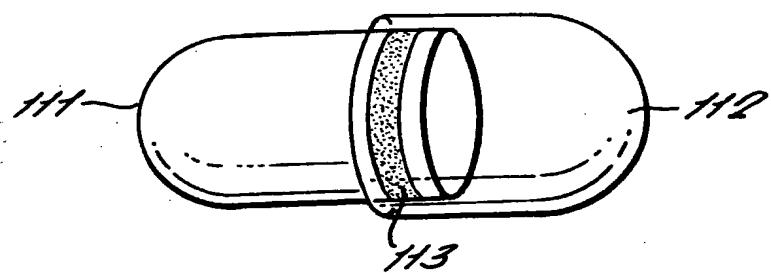


FIG. 10A.

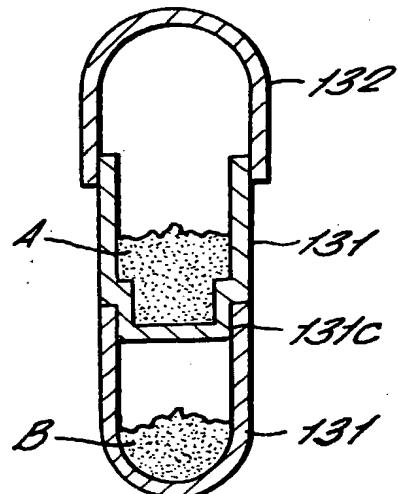
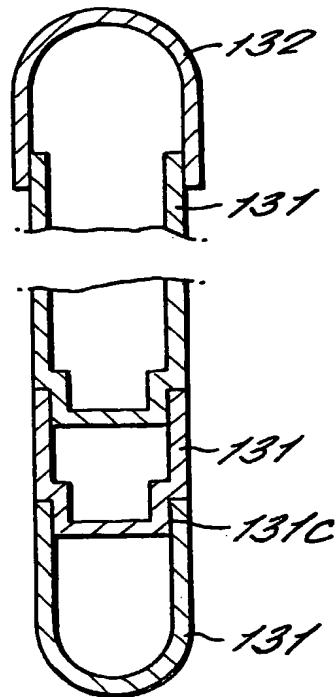


FIG. 10B.



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FIG. 11A.

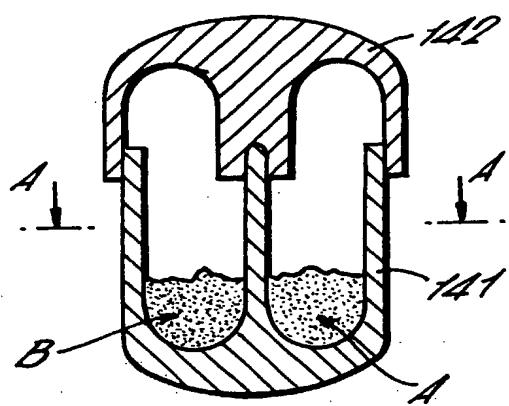


FIG. 11B.

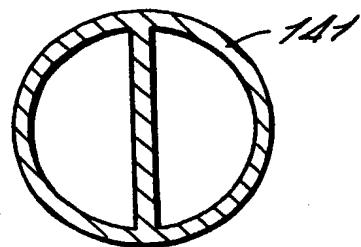
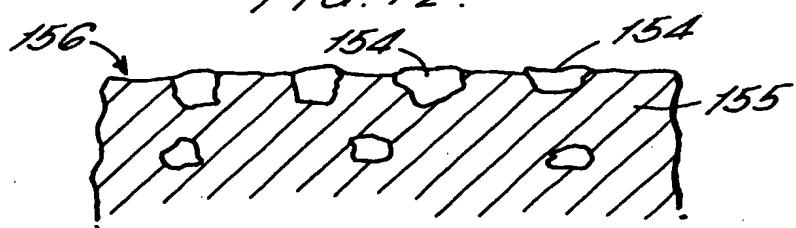
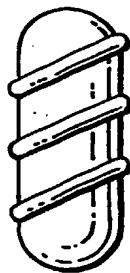
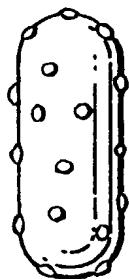
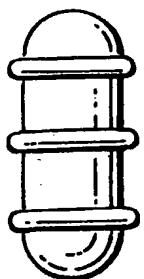
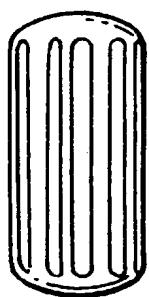
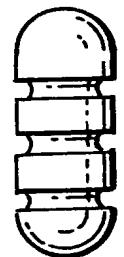
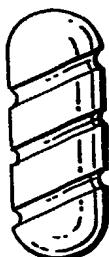
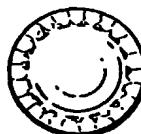


FIG. 12.



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FIG. 13A. FIG. 13B. FIG. 13C. FIG. 13D. FIG. 13E.*FIG. 13F.**FIG. 13H.**FIG. 13J.**FIG. 13K.**FIG. 13L.**FIG. 13M.*

INTERNATIONAL SEARCH REPORT

Application No
PCT/GB 00/04376A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 B65D65/46

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 B65D

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	EP 0 549 803 A (JUJO) 7 July 1993 (1993-07-07) page 5, line 12-19 ---	1-67
A	EP 0 941 939 A (KEMTEC) 15 September 1999 (1999-09-15) column 3, line 40-48; figures 1-3 ---	1-67
A	EP 0 244 084 A (BALL) 4 November 1987 (1987-11-04) column 7, line 13-20; figures 1-4 ---	1-67
A	WO 95 23099 A (CIBA-GEIGY) 31 August 1995 (1995-08-31) page 3, line 12 -page 5, line 5 ---	1-67
		-/-

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Date of mailing of the international search report

20 February 2001

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Name and mailing address of the ISA
European Patent Office, P.B. 5818 Patentlaan 2
NL - 2280 HV Rijswijk
Tel. (+31-70) 340-2040, Tx. 31 651 epo nl,
Fax: (+31-70) 340-3016

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INTERNATIONAL SEARCH REPORT

In nat Application No
PCT/GB 00/04376

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 97 08244 A (KWIZDA GMBH) 6 March 1997 (1997-03-06) page 3, line 1-8 -page 8, line 18-26 ---	1-67
A	WO 97 00282 A (ZENECA) 3 January 1997 (1997-01-03) page 2, line 10-24 ---	25
A	US 4 348 293 A (CLARKE) 7 September 1982 (1982-09-07) column 4, line 61-67 ---	36
A	GB 650 961 A (GOURGUES) 7 March 1951 (1951-03-07) page 1, line 81-85; figure 5 ---	36
A	DE 37 11 577 A (DOLDERER) 27 October 1988 (1988-10-27) column 1, line 36 -column 2, line 9 ---	53,54
A	EP 0 578 277 A (SCHMITZ-SCHOLL) 12 January 1994 (1994-01-12) page 1; figures 1,2 ---	1-67
A	WO 95 15249 A (HADLEY) 8 June 1995 (1995-06-08) page 1-7; figures 1-5 ---	1-67
A	US 3 275 448 A (SOMMER) 27 September 1966 (1966-09-27) column 1-4; figures 1-5 ---	1-67
A	US 1 592 395 A (SULZBERGER) 13 July 1926 (1926-07-13) page 1-2; figures 1-4 -----	1-67

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/GB 00/04376

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
EP 549803	A 07-07-1993	AU 659171 B AU 2000392 A CA 2090016 A WO 9300476 A JP 3053431 B		11-05-1995 25-01-1993 21-12-1992 07-01-1993 19-06-2000
EP 941939	A 15-09-1999	NONE		
EP 244084	A 04-11-1987	NONE		
WO 9523099	A 31-08-1995	AU 1757095 A BR 9506868 A DE 69509075 D DE 69509075 T EP 0746514 A IL 112672 A JP 9509125 T ZA 9501522 A		11-09-1995 09-09-1997 20-05-1999 14-10-1999 11-12-1996 15-06-1998 16-09-1997 08-12-1995
WO 9708244	A 06-03-1997	AT 175432 T AU 6865196 A CA 2230382 A DE 19680737 D DE 59601120 D DK 846145 T EP 0846145 A ES 2128182 T SI 846145 T		15-01-1999 19-03-1997 06-03-1997 25-11-1999 18-02-1999 30-08-1999 10-06-1998 01-05-1999 30-06-1999
WO 9700282	A 03-01-1997	AU 5827696 A EP 0835278 A JP 11507971 T		15-01-1997 15-04-1998 13-07-1999
US 4348293	A 07-09-1982	AR 229571 A AU 534223 B AU 5292379 A BR 7908913 A CA 1130694 A EP 0011502 A ES 486067 D ES 8101639 A WO 8001078 A JP 60024838 B JP 55500902 T MX 151028 A ZA 7906189 A		30-09-1983 12-01-1984 22-05-1980 21-07-1981 31-08-1982 28-05-1980 16-12-1980 16-03-1981 29-05-1980 14-06-1985 06-11-1980 11-09-1984 24-06-1981
GB 650961	A	NONE		
DE 3711577	A 27-10-1988	NONE		
EP 578277	A 12-01-1994	DE 3921584 A AT 101834 T CA 2019672 A DE 8915452 U DE 59004659 D DK 405146 T EP 0405146 A		10-01-1991 15-03-1994 31-12-1990 19-07-1990 31-03-1994 28-03-1994 02-01-1991

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No
PCT/GB 00/04376

Patent document cited in search report	Publication date	Patent family member(s)		Publication date
EP 578277	A	ES	2050300 T	16-05-1994
		JP	3133760 A	06-06-1991
WO 9515249	A	08-06-1995	AU 1194495 A	19-06-1995
US 3275448	A	27-09-1966	NONE	
US 1592395	A	13-07-1926	NONE	